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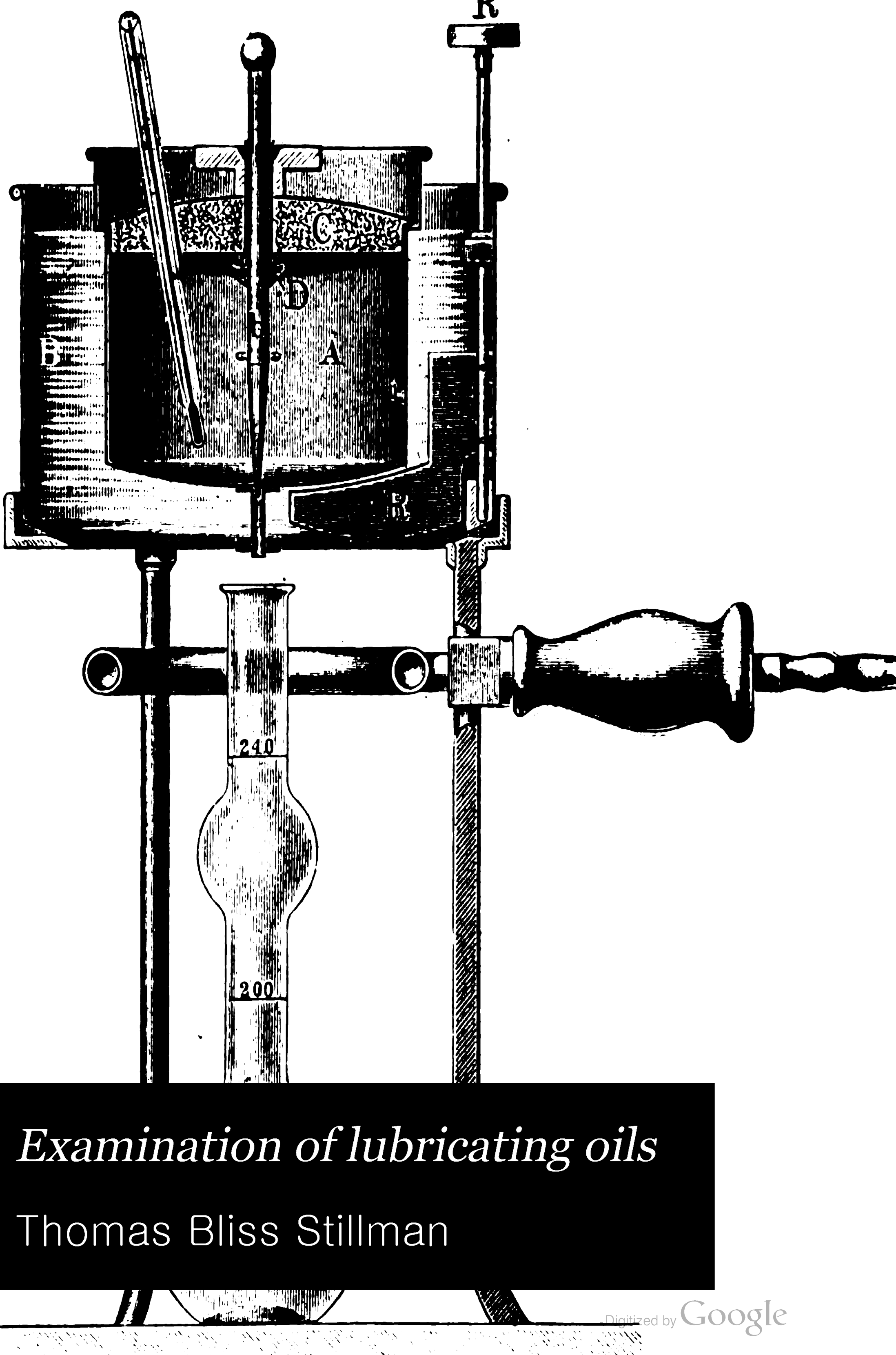

Examination of lubricating oils

Thomas Bliss Stillman

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THE EXAMINATION OF LUBRICATING OILS

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Examination of Lubricating Oils

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PREFACE.

This manual of the testing of lubricating oils is the result of many years of experience in the subject.

The rapid advancement in various machine construction requiring corresponding varieties of oils for lubrication has complicated the testing of methods somewhat.

This is especially true of the automobile, and cold storage specifications for lubricating oils.

The article entitled "Apparatus for the Examination and Study of the Behavior of Valve and Cylinder Oils and other Petroleum and Lubricating Oils in Saturated and Superheated Steam, Carbon Dioxide and Other Gases," by P. H. Conradson, chief chemist of the Galena-Signal Oil Co., Franklin, Pa., is found on pages 114–119 and is recommended as an experiment of great value on this subject by a more practical experience in the testing of lubricating oils.

Aug. 25, 1914,
Laboratory of Engineering Chemistry,
Jersey City, N. J.

321447

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The Examination of Lubricating Oils

The generally accepted conditions of a good lubricant are as follows:

1. Body enough to prevent the surfaces, to which it is applied, from coming in contact with each other.
2. Freedom from corrosive acids, either of mineral, animal, or vegetable origin.
3. As fluid as possible, consistent with "body."
4. A minimum coefficient of friction.
5. High "flash" and "burning" points.
6. Freedom from all materials liable to produce oxidation or "gumming," or addition of "artificial thickeners."
7. Must not be easily thinned or vaporized by heat or thickened by cold.

The examinations to be made to verify the above are both chemical and mechanical, and are usually arranged in the following order:

1. Specific gravity.
2. Cold test.
3. Viscosity.
4. Iodine absorption.
5. Flash and fire tests.
6. Acidity.
7. Maumené's test.
8. Identification of the oil, whether a simple mineral oil, animal oil, vegetable oil, or a mixture.
9. Coefficient of friction.

1. Specific Gravity

In the chemical laboratory the hydrometers used are generally marked with the specific gravity direct. In the oil trade, however, and in general commercial practice the Baumé hydrometer is used, and the following precaution is necessary.

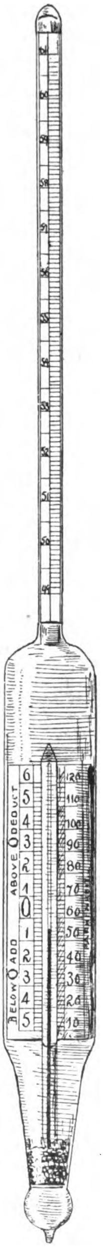

Fig. 1.

If the oil is not liquid enough to flow easily, it must be warmed until so, and then tested with the hydrometer. The latter should move easily and freely in the liquid. As all specific gravities are comparable at 60° F., it will be necessary to make correction for temperature; if the temperature of the oil is above 60° F., the readings of the hydrometer are too large; if below 60° F., the readings are too small. Suppose an oil registers 28° Baumé at 72° F., we make use of the table on page 5 and find the corrected reading to be 27.2° Baumé.

To convert this into specific gravity the following table is used:

For Liquids Lighter than Water $\text{sp. gr.} = \dfrac{140}{130 + {}^\circ\text{Bé}}$ at 60° F.

°Bé.	Sp. gr.	°Bé.	Sp. gr.	°Bé.	Sp. gr.	°Bé.	Sp. gr.
10	1.0000	28	0.8861	46	0.7955	64	0.7216
11	0.9929	29	0.8805	47	0.7910	65	0.7179
12	0.9859	30	0.8750	48	0.7865	66	0.7143
13	0.9790	31	0.8696	49	0.7821	67	0.7107
14	0.9722	32	0.8642	50	0.7778	68	0.7071
15	0.9655	33	0.8589	51	0.7735	69	0.7035
16	0.9589	34	0.8537	52	0.7692	70	0.7000
17	0.9524	35	0.8485	53	0.7650	71	0.6965
18	0.9459	36	0.8434	54	0.7609	72	0.6931
19	0.9396	37	0.8383	55	0.7568	73	0.6897
20	0.9333	38	0.8333	56	0.7527	74	0.6863
21	0.9272	39	0.8284	57	0.7487	75	0.6829
22	0.9211	40	0.8235	58	0.7447	76	0.6796
23	0.9150	41	0.8187	59	0.7407	77	0.6763
24	0.9091	42	0.8140	60	0.7368	78	0.6731
25	0.9032	43	0.8092	61	0.7330	79	0.6699
26	0.8970	44	0.8046	62	0.7292	80	0.6667
27	0.8917	45	0.8000	63	0.7254	90	0.6363
						100	0.6086

and we find that 27.2° Baumé is equal to 0.8906 specific gravity.[1]

---

[1] If we assume the standard temperature for this purpose to be 15.5°C, the gravity of any oil at higher or lower temperature can be calculated from the following formula:

$$G = G' + K\,(T - 15.5^\circ),$$

in which G is the specific gravity at 15.5°, G′ the specific gravity at T, T = temp. of room, and K a factor varying with the different oils, as follows:

Factor for Calculating Specific Gravity of Oils[2]

	Correction for 1°C.
Cod liver oil	0.000646
Olive oil	0.000629
Rape oil	0.000620
Lard oil	0.000658
Peanut oil	0.000655
Cottonseed oil	0.000629
Corn oil	0.000630
Sesame oil	0.000624

[2] A. E. Leach, "Food Inspection and Analysis." p. 372, gives the formula for ordinary use as $G = G' + 0.00064\,(T - 15.5^\circ\text{C})$.

Fig. 1 represents a Tagliabue hydrometer for oils; it contains a thermometer, also a scale to make the readings at 60° F. Subtract 1° Baumé for every 10° F. above 60° F., and add 1° Baumé for every 10° F. below 60° F.

Thus, if the hydrometer, when placed in the oil, reads 26° Baumé and the temperature of the oil 80° F., the correct reading will be 24.7° Baumé at 60° F. The specific gravity test is an important one; by it an admixture of certain oils with mineral oil is indicated. For instance, a lubricating oil of specific gravity 0.915 was found by qualitative analysis to be composed of mineral oil and menhaden oil. Knowing the kinds of oil composing the mixture, an approximation of the per cents. would be obtained as follows (page 6):

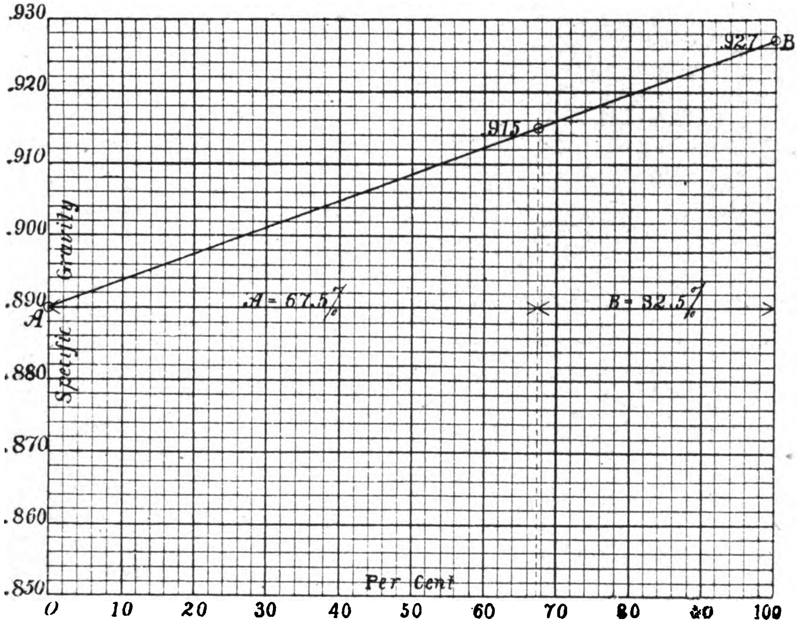

Fig. 2.

TABLE OF SPECIFIC GRAVITY WITH CORRECTION FOR TEMPERATURE (LIQUIDS LIGHTER THAN WATER).

Hydrometer.	40° F.	42° F.	44° F.	46° F.	48° F.	50° F.	52° F.	54° F.	56° F.	58° F.	60° F.	62° F.	64° F.	66° F.	68° F.	70° F.
20° B.......	21.1	21.0	20.9	20.7	20.6	20.5	20.4	20.3	20.2	20.1	20.0	19.9	19.8	19.6	19.5	19.4
22° B.......	23.2	23.0	22.9	22.8	22.7	22.5	22.4	22.3	22.2	22.1	22.0	21.9	21.7	21.6	21.5	21.4
24° B.......	25.2	25.1	25.0	24.8	24.7	24.6	24.5	24.4	24.3	24.1	24.0	23.9	23.7	23.6	23.5	23.4
26° B.......	27.3	27.2	27.0	26.9	26.7	26.6	26.5	26.4	26.3	26.1	26.0	25.9	25.7	26.6	25.5	25.3
28° B.......	29.3	29.2	29.1	28.9	28.8	28.6	28.5	28.4	28.3	28.1	28.0	27.9	27.8	27.6	27.5	27.3
30° B.......	31.4	31.2	31.1	31.0	30.8	30.7	30.5	30.4	30.3	30.1	30.0	29 9	29.7	26.6	29.5	29.3
32° B.......	33.5	33.3	33.1	33.0	32.9	32.7	32.6	32.4	32.3	32.1	32.0	31.9	31.7	31.6	31.4	31.3
34° B.......	35.5	35.4	35.2	35.1	34.9	34.8	34.6	34.5	34.3	34.2	34.0	33.8	33.7	33.5	33.4	33.3
36° B.......	37.6	37.4	37.2	37.1	36.9	36.8	36.6	36.5	36.3	36.2	36.0	35.8	35.7	35.5	35.4	35.2
38° B.......	39.6	39.4	39.3	39.1	3.90	38.8	38.6	38.5	38.3	38.2	38.0	37.8	37.7	37.5	37.4	37.2
40° B.......	41.6	41.5	41.3	41.2	41.0	40.8	40.7	40.5	40.3	40.2	40.0	39.8	39.7	39.5	39.4	39.2
42° B.......	43.7	43.5	43.4	43.2	43.0	42.9	42.7	42.5	42.3	42.2	42.0	41.8	41.7	41.5	41.3	41.2
44° B.......	45.7	45.6	45.4	45.2	45.1	44.9	44.7	44.5	44.4	44.2	44.0	43.8	43.7	43.5	43.3	43.1
46° B.......	47.8	47.6	47.4	47.3	47.1	46.9	46.7	46.6	46.4	46.2	46.0	45.8	45.7	45.5	45.3	45.1
48° B.......	49.9	49.7	49.5	49.3	49.1	48.9	48.8	48.6	48.4	48.2	48.0	47.8	47.6	47.4	47.3	47.1
50° B.......	52.0	51.8	51.6	51.4	51.2	51.0	50.8	50.6	50.4	50.2	50.0	49.8	49.6	49.4	49.2	49.1
52° B.......	54.0	53.8	53.6	53.4	53.2	53.0	52.8	52.6	52.4	52.2	52.0	51.8	51.6	51.4	51.1	51.0
56° B.......	58.2	58.0	57.8	57.5	57.3	57.1	56.9	56.7	56.4	56.2	56.0	55.8	55.6	55.4	55.2	55.0
58° B.......	60.3	60.0	59.8	59.6	59.3	59.1	58.9	58.7	58.4	58.2	58.0	57.8	57.6	57.3	57.1	56.9
60° B.......	62.3	62.1	61.9	61.6	61.4	61.1	60.9	60.7	60.5	60.2	60.0	59.8	59.6	59.3	59.1	58.9
64° B.......	66.5	66.2	66.0	65.7	65.5	65.2	65.0	64.7	64.5	64.2	64.0	63.8	63.5	63.3	63.1	62.8
66° B.......	68.6	68.3	68.0	67.8	67.5	67.2	67.0	66.8	66.5	66.3	66.0	65.7	65.5	65.3	65.0	64.8
70° B.......	72.7	72.5	72.2	71.9	71.6	71.3	71.7	70.8	70.5	70.3	70.0	69.7	69.5	69.2	69.0	68.7
72° B.......	74.8	74.5	74.2	73.9	73.6	73.3	73.1	72.8	72.5	72.3	72.0	71.7	71.5	71.2	70.9	70.7
76° B.......	78.9	78.6	78.3	78 0	77.7	77.4	77.1	76.9	76.6	76.3	76.0	75.7	75.4	75.2	74.9	74.6
80° B.......	83.0	82.7	82.4	82.1	81.8	81.5	81.2	80.9	80.6	80.3	80.0	79.7	79.4	79.1	78.8	78.5

TABLE OF SPECIFIC GRAVITY WITH CORRECTION FOR TEMPERATURE (LIQUIDS LIGHTER THAN WATER).—(*Continued*).

Hydrometer.	72° F.	74° F.	76° F.	78° F.	80° F.	82° F.	84° F.	86° F.	88° F.	90° F.	92° F.	94° F.	96° F.	98° F.	100° F.
20° B.............	19.3	19.2	19.1	19.0	18.9	18.7	18.7	18.6	18.5	18.4	18.2	18.1	18.0	17.8	17.9
22° B.............	21.3	21.1	21.0	20.9	20.8	20.7	20.6	20.5	20.4	20.3	20.2	20.1	19.9	19.8	19.7
24° B.............	23.2	23.1	23.0	22.9	22.8	22.6	22.5	22.4	22.3	22.2	22.1	21.9	21.8	21.7	21.6
26° B.............	25.2	25.1	25.0	24.8	24.7	24.6	24.5	24.4	24.2	24.1	24.0	23.9	23.7	23.6	23.5
28° B.............	**27.2**	**27.1**	**26.9**	**26.8**	**26.7**	**26.5**	**26.4**	**26.3**	**26.2**	**26.0**	**25.9**	**25.8**	**25.6**	**25.5**	**25.4**
30° B.............	29.2	29.0	28.9	28.8	28.6	28.5	28.4	28.2	28.1	28.0	27.8	27.7	27.6	27.5	27.3
32° B.............	31.2	31.0	30.9	30.7	30.6	30.4	30.3	30.2	30.0	29.9	29.7	29.6	29.5	29.3	29.2
34° B.............	33.1	36.0	32.8	32.7	32.5	32.4	32.2	32.1	32.0	31.8	31.6	31.5	31.4	31.2	31.1
36° B.............	35.1	34.9	34.8	34.6	34.5	34.3	34.2	34.0	33.9	33.7	33.5	33.4	33.3	33.1	33.0
38° B.............	**37.1**	**36.9**	**36.8**	**36.6**	**36.4**	**36.3**	**36.1**	**36.0**	**35.8**	**35.6**	**35.5**	**35.3**	**35.2**	**35.0**	**34.9**
40° B.............	39.0	38.9	38.7	38.5	38.4	38.2	38.1	37.9	37.7	37.6	37.4	37.2	37.1	36.9	36.8
42° B.............	41.0	40.8	40.7	40.5	40.3	40.2	40.0	39.8	39.7	39.5	39.3	39.2	39.0	38.9	38.7
44° B.............	43.0	42.8	42.6	42.5	42.3	42.1	42.0	41.8	41.6	41.4	41.3	41.1	40.9	40.8	40.6
46° B.............	44.9	44.8	44.6	44.4	44.2	44.1	43.9	43.7	43.6	43.4	43.1	43.0	42.9	42.7	42.6
48° B.............	**46.9**	**46.7**	**46.5**	**46.4**	**46.2**	**46.0**	**45.8**	**45.7**	**45.5**	**45.3**	**45.2**	**45.0**	**44.8**	**44.6**	**44.5**
50° B.............	48.9	48.7	48.5	58.3	48.1	48.0	47.8	47.6	47.4	47.3	47.1	46.9	46.7	46.5	46.4
52° B.............	50.8	50.6	50.4	50.3	50.1	49.9	49.7	49.5	49.4	49.2	49.0	48.8	48.6	48.4	48.3
56° B.............	54.8	54.5	54.3	54.1	53.9	53.7	53.5	53.4	53.2	53.0	52.8	52.6	52.4	52.2	52.0
58° B.............	36.7	56.5	56.3	56.1	55.9	55.7	55.5	55.3	55.1	54.9	54.7	54.5	54.3	54.1	53.9
60° B.............	**58.7**	**58.5**	**58.2**	**68.0**	**57.8**	**57.6**	**57.4**	**57.2**	**57.0**	**56.8**	**56.6**	**56.4**	**56.2**	**55.9**	**55.7**
64° B.............	62.6	62.4	62.1	61.9	61.7	61.4	61.2	61.0	60.8	60.6	60.4	60.2	59.9	59.7	59.5
66° B.............	64.5	64.3	64.1	63.8	63.6	63.4	63.2	62.9	62.7	62.5	62.3	62.1	61.8	61.6	61.4
70° B.............	68.4	68.2	68.0	67.7	67.5	67.2	67.0	66.7	66.5	66.3	66.1	65.8	65.6	65.3	65.1
72° B.............	70.4	70.2	69.9	69.6	69.4	69.2	68.9	68.7	68.4	68.2	67.7	67.7	67.4	67.2	67.0
76° B.............	74.3	74.0	73.8	73.5	73.2	73.0	72.7	72.5	72.2	71.9	71.7	71.4	71.2	70.9	70.7
80° B.............	78.2	77.9	77.6	77.3	77.1	76.8	76.5	76.3	76.0	75.7	75.2	75.1	74.9	74.6	74.4

Mineral oil .... **Specific gravity** = 0.890 (B)
Menhaden oil .... **Specific gravity** = 0.927 (A)
Specific gravity of mixture .... = 0.915 (M)

$$\text{Let } A - M = C. \quad (0.927 - 0.915 = 0.012)$$
$$M - B = D. \quad (0.915 - 0.890 = 0.025)$$

$$\text{Then } \frac{D}{C + D} = \text{per cent. of A } \left(\frac{0.025}{0.037}\right)$$

and

$$\frac{C}{C + D} = \text{per cent. of B } \left(\frac{0.012}{0.037}\right).$$

The result being

	Per cent.
Menhaden oil	67.5
Mineral oil	32.5

A more rapid method is shown graphically, thus: in Fig. 2 let the abscissas represent per cents. and the ordinates the specific gravities. From the point indicated (on the line AB) 0.915 the specific gravity of the mixture the per cents. are read on abscissa line 67.5 for A and 32.5 per cent. for B.

Another instrument used for the determination of the specific gravity of oils is the Westphal balance, as improved by Williams.[1]

This apparatus (Fig. 3) is very accurate and should be used as a check determination of the gravity made by the hydrometer.

Directions for Using the Williams-Westphal Balance

To Determine the Specific Gravity of Liquids:—Hang thermometer plummet upon hook and after leveling the instrument, bring beam to equilibrium in air by turning the adjusting weight on threaded portion of the beam. It is desirable to verify this adjustment by immersing thermometer plummet in distilled water at 15° C. and hanging one of the largest horse-shoe weights upon the hook; this should exactly restore the beam to equilibrium. Care should be taken that the thermometer plummet be fully immersed throughout the complete swing of the beam. After wiping plummet it is immersed in the solution to be tested, this having been brought to the proper temperature, and weights added, beginning with largest until equilibrium of the beam is restored. If the solution is heavier than water one of the largest weights must hang on the hook at the end of the beam. In reading the weights, the large weight at the end of the beam indicates one, the other large weight indicates tenths, corresponding to the position it occupies on the graduated portion of the beam, the next smaller weight indicates hundredths, the next smaller thousandths and the smallest ten-thousandths.

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[1] "For Determination of the Spec. Grav. of Liquids and Solids, both Soluble and Insoluble in Water."

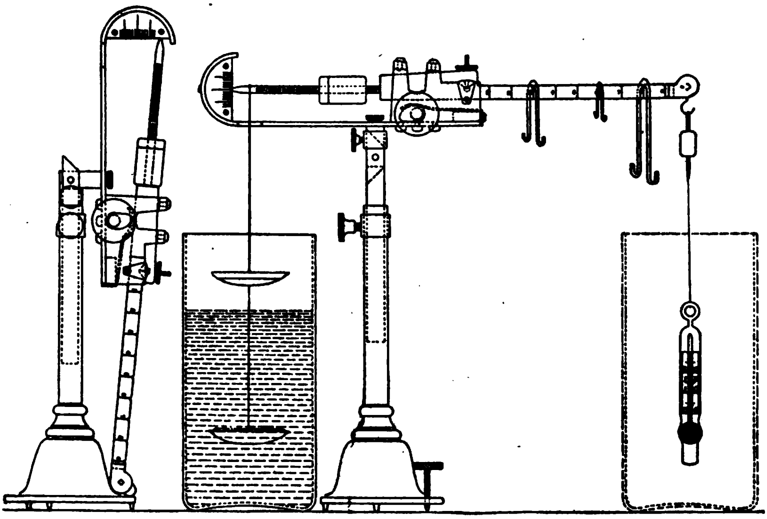

Fig. 3.

To Determine the Specific Gravity of Solids Insoluble in Water:—The plummet is first wiped dry, the pan hanger is attached at the threaded end of the beam, and water at 15° C. is added until the lower pan is immersed throughout the complete swing of the beam. The beam is now adjusted to equilibrium by turning the weight on threaded portion of the beam. The substance is now placed in the upper pan and the horse-shoe weights added as above until equilibrium is restored. This gives weight of substance in air. The substance if in the form of a fragment is now transferred to the lower pan, if in the form of a powder the upper pan with its contents is transferred to the lower position, and after wiping lower pan it is transferred to the upper position. Weights are now added until equilibrium is restored, this reading giving weight of substance in water. Calculations:—

$$\frac{\text{Weight in air}}{\text{Weight in air} - \text{Weight in water}} = \text{Specific Gravity.}$$

To Determine the Specific Gravity of a Substance Acted Upon by Water:—For example, Portland Cement. Select some liquid such as carbon tetrachloride, carbon bi-sulphide or benzine, which has no action upon the substance. Immerse lower pan in this liquid, and after adjusting beam to equilibrium, determine weight in air and weight in this liquid as above, which will give the apparent specific gravity of the substance.

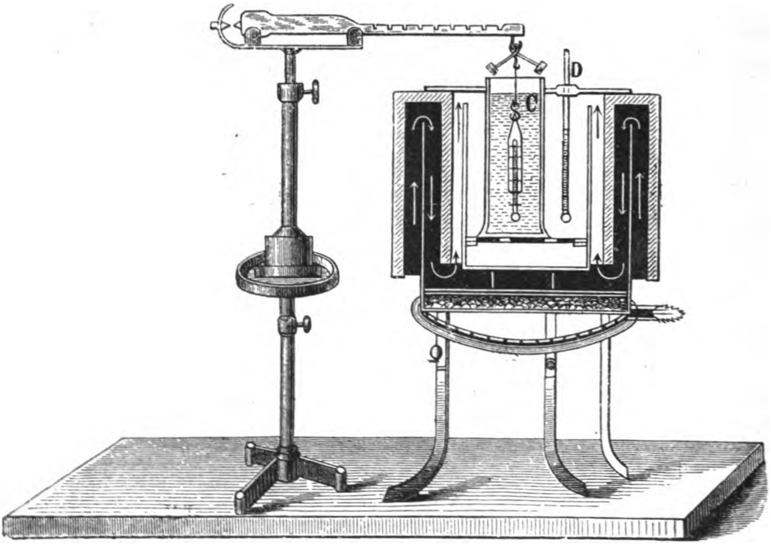

Fig. 4.

To Obtain True Specific Gravity:—The specific gravity of the liquid used is determined with thermometer plummet and the apparent specific gravity multiplied by specific gravity of liquid used, gives the true specific gravity of substance.

If oil is too thick, at ordinary temperatures, for the determination of the gravity, it should be heated sufficiently and the modified Westphal balance (Fig. 4) used.

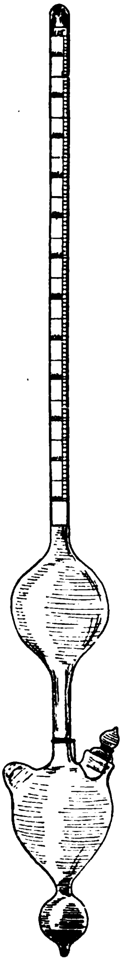

Fig. 5.

If only small amounts of the oil are obtainable a small picnometer, or an Aræo-picnometer of Eichhorn can be used. This invention is described by Dr. H. Hensoldt, of the Petrographical Laboratory of Columbia College, New York, in the *Scientific American Supplement* of March 21, 1891, with a drawing. The important feature of this instrument consists in a small glass bulb (attached to the spindle), which is filled with the liquid whose gravity is to be taken. Thus instead of floating the entire apparatus in the test fluid, only a very small quantity of the latter is required.

The glass bulb, when filled with the test fluid, is closed by means of an accurately fitting glass stopper, and the instrument is then placed in a glass cylinder filled with distilled water at 17.5° C. (Fig. 5).

The gravity is then at once shown on the divided scale in upper portions of the spindle.

The following table converts degrees of the various hydrometers into specific gravity. (Liquids lighter than water.)

$$\text{Gay Lussac, } 4^\circ\text{C.} = \frac{100}{100 + n} = \text{specific gravity.}$$

$$\text{Beck, } 12.5^\circ\text{ C.} = \frac{170}{170 + n} = \text{specific gravity.}$$

$$\text{Cartier, } 12.5^\circ\text{ C.} = \frac{136.8}{126.1 + n} = \text{specific gravity.}$$

$$\text{Baumé hydrometer at } 15^\circ\text{ C.} = \frac{140}{130 + n} = \text{specific gravity.}$$

$$\left.\begin{array}{l}\text{Brix hydrometer, Fisher}\\ \text{hyrometer at } 15.6^\circ\text{ C.}\end{array}\right\} = \frac{400}{400 + n} = \text{specific gravity.}$$

n = degrees indicated upon the spindle.

TABLE OF SPECIFIC GRAVITY OF OILS USED WITH MINERAL OILS FOR LUBRICATING PURPOSES.

	Sp. Gr.
Sperm oil	0.883
Olive oil	0.916
Cotton-seed oil (white)	0.925
Cotton-seed oil (brown)	0.930
Castor oil	0.960
Mineral oil	0.860 to 0.925
Dolphin oil	0.922
Neat's foot oil	0.915
Lard oil	0.915
Tallow oil	0.903
Menhaden oil	0.928
Rape-seed oil	0.916
Rosin oil	0.980 to 1.05
Blown oils, made by oxidation of rape-seed oil, cotton-seed oil, etc	0.930 to 0.970
Corn oil	0.922

2. The Cold Test

The degree at which an oil becomes semi-solid and refuses to flow freely is considered the cold test and is performed as follows:

Fifty cc. of the oil are transferred to a narrow bottle (capacity 100 cc.), stoppered with a rubber stopper, through which is inserted a thermometer, the bulb of which reaches an inch or more into the oil.

The bottle is placed in a mixture of ice and salt, or other freezing compound, and retained there until the oil becomes solid. It is then removed and allowed to warm until the contents become somewhat thinner in consistence. The bottle is inclined from side to side until the oil begins to flow, when the temperature is taken.

At this particular temperature the oil is neither at its normal fluidity, nor is it solid, and while this method does not correctly indicate the exact temperature of the solidifying-point, it does show the point at which the oil ceases to flow readily, the important one to the oil inspector.

In lubricating oils, to be used in railroad practice, this cold test is a vital one, and receives in the laboratories of the different railroads of the United States considerable attention.

A mineral lubricating oil, non-paraffine, of good quality, does not show any material difference in its consistency at 25° C. or 10° C., but a radical change would be indicated at 10° C. if some of the animal or vegetable oils were a component.

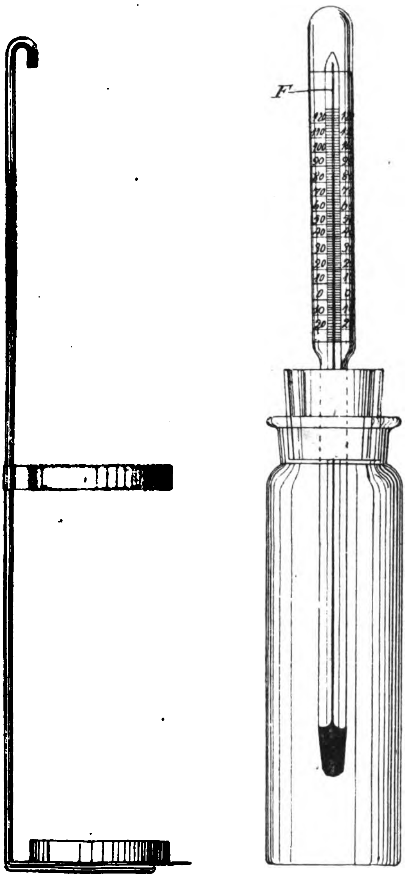

Fig. 6.

Fig. 6 represents the glass apparatus with the thermometer arranged for the cold test, and is surrounded by any mixture capable of producing the required degree of cold.

The following determinations of the cold test, made in my laboratory, will show the wide range in this regard between many of the oils, used in lubrication:

	Degrees F.
Elain oil	42.8
Saponified red oil	41.
Prime Neat's foot oil	24.8
White Neat's foot oil	....
Pure hoof oil	42.8
Prime lard oil	44.6
No. 1 Lard oil	44.6
XXX Lard oil	37.4
American sod oil	33.8
English sod oil	75.
Tallow oil	79.
Dog fish oil	19.
Right whale oil (Pacific)	32.
Unbleached bowhead whale oil (Pacific)	19.
Bleached whale oil (Pacific)	8.6
Natural sperm oil (Pacific)	32.
Bleached sperm oil (Pacific)	24.8
Herring oil (Pacific)	32.
Natural winter sperm oil (Atlantic)	30.2
Bleached winter sperm oil (Atlantic)	24.8
Natural spring sperm oil (Atlantic)	50.
Bleached spring sperm oil (Atlantic)	46.
Natural winter whale oil (Atlantic)	28.
Bleached winter whale oil (Atlantic)	23.
Natural spring whale oil (Atlantic)	41.
Bleached spring whale oil (Atlantic)	35.6
Prime crude menhaden oil	24.8
Brown strained menhaden oil	19.5
Light strained menhaden oil	19.5
Natural winter menhaden oil	16.
Bleached winter menhaden oil	10.4
Extra bleached winter white menhaden oil	12.
Bank oil	24.8
Straits oil	19.5
Sea elephant oil	41.
Black fish oil	17.6
Rosin oil, 1st run	37.4
Rosin oil, 2d run	— 2.2
Rosin oil, 3d run	— 4.
Castor oil	— 0.4
Crude cotton-seed oil	19.4
Prime summer yellow cotton-seed oil	23.
Off quality summer yellow cotton-seed oil	21.
Prime quality winter cotton-seed oil	14.

	Degrees F.
Off quality winter cotton-seed oil	17.6
Prime quality summer white cotton-seed oil	26.6
Off quality summer white cotton-seed oil	17.6
Prime quality winter white cotton-seed oil	15.8
Off quality winter white cotton-seed oil	23.
No. 1 French Degras oil	77.
No. 2 French Degras oil	77.
English Degras oil	64.4
Olive Oil	37.4
Oleo oil	75.

The method in use by the Motive Power Department of the P. R. R. for determination of the *cold test* and the *chilling points* of oils is as follows:

For cold test, put about 1 oz. of the liquid to be tested into a common 4 oz. sample bottle, and place a short, stout thermometer in it. Then place the bottle in a situation where the liquid will become frozen, using for this purpose a freezing mixture if necessary. When the liquid has become solid throughout, remove from the cold and allow the liquid to soften, thoroughly stirring and mixing it at the same time, by means of the thermometer, until the mass will run from one end of the bottle to the other. Grasp the bottle by the neck, having in the same hand a little waste or towel, which encloses the thermometer, withdraw the thermometer through the waste, to wipe it far enough to see the mercury, and read the temperature. The reading is the cold test of the liquid.

For chilling point, where it is desired to know *whether the liquid remains clear at any given temperature,* use the same bottle, amount of liquid and thermometer as for cold test. Expose the liquid to the given temperature, or a little below, stir it with the thermometer until the temperature of the whole liquid becomes that desired, then allow to stand for the time specified, at the end of which observe whether the liquid remains transparent and free from flocks or scales of congealed constituents. If so, it stands test.

For chilling point, where it is desired to know *at what temperature flocks or scales of congealed constituents of more or less* *opacity takes place.* Use bottle, amount of liquid, and thermometer as before. Expose to a temperature 5° colder than that at which the liquid is perfectly clear and free from flocks or scales, and allow the temperature to fall to that of the source of cold, with constant watching and occasional stirring. Lower the source of cold 5° more and treat as before. Proceed in this way until *opacity* or flocks or scales begin to show. The reading of the thermometer when this is the case is the temperature sought.

Freezing mixtures. For cold test. If the cold test is above 35° F., use a mixture of snow or pounded ice and water in any suitable non-conducting vessel. The bottle with oil and thermometer is plunged into the liquid and allowed to remain until the liquid in the bottle is frozen solid. For cold tests from 35° F. down to zero F., use a mixture of two parts, by weight, of snow or pounded ice, and one part of common salt.

For cold test from zero F. to 30° F. below use a mixture of three parts, by weight, of crystallized calcium chloride and two parts of snow or very finely pounded ice. The crystallized calcium chloride, if obtained in the market, should be crushed in a mortar to pieces not larger than wheat kernels. For cold tests approaching the lower range of the above limit, the vessel should be non-conducting and should be cooled to 32° F. before putting in the calcium chloride and ice. The crystallized calcium chloride may be bought in the market, but the following method gives a very cheap and efficient article from materials always at hand in every good laboratory—viz.: Put abount a pound of pulverized carbonate of lime—marble dust works best—into a casserole holding three pints or two quarts, and pour concentrated commercial hydrochloric acid on it, little at a time. A neutral solution of calcium chloride is formed very quickly, after each addition of acid, and in a short time the mass becomes wet enough to be stirred to facilitate the action. Proceed with the addition of the acid until nearly all the marble dust is dissolved, taking care to avoid an excess of acid. Pouring the acid on the marble dust, instead of adding the dust to the acid, avoids the suffocating fumes of HCl, which are sure to be given off if the latter procedure is followed. After the last addition of acid, filter into any convenient metal vessel whose weight is known, then boil until a drop of the liquid put on a watch glass, and cooled by placing the glass on a lump of ice, becomes solid moderately quickly, then cool by placing the vessel in another vessel containing ice and water. As the temperature falls, the calcium chloride should be stirred to prevent the formation of a compact mass not easy to break up. If the operation is properly performed, when the temperature of the calcium chloride reaches 40° F. or a little below, the material in the metal vessel will be a more or less mushy mass of crystals of calcium chloride. Remove now the metal vessel from the ice water, wipe it dry on the outside, and weigh to determine the amount of calcium chloride. Wrap the bottom and sides of this vessel well with towels or other non-conducting material, and then for every three parts of calcium chloride add two parts of snow or finely pounded ice, and stir thoroughly. After a minute or two the material to be tested can be placed in the liquid, and the whole thing should then be covered to prevent access of heat.

For chilling point, when it is desired to know whether a liquid remains clear at any given temperature. Temperatures from any one at which the liquid to be tested is clear down to 32° F. are easily obtained. Put a gallon of water in a bucket made of wood or indurated fiber, and add warm or colder water or ice, as the case may require, until the desired temperature is obtained, then add the bottles of liquid to be tested, taking care to keep the water the required temperature during the cooling of the liquids in the bottles by the proper additions. Temperatures from 32° down to zero F. may also be easily obtained. To a gallon of water in a bucket of wood or indurated fiber, add 15 lbs. of cracked ice. Stir thoroughly with a wooden stick, and when the temperature has reached 32° F. or thereabouts, add, with continued stirring, dry common salt, sufficient to produce the temperature desired. Approximately each quarter pound of salt added will lower the temperature under the conditions given above 2° F., until about 10° is reached, when twice the quantity must be added to bring the temperature down 2°. As the usual specified temperatures at which chilling points are observed are 32°, 20°, 10°, and zero F., it is fairly safe to say that these temperatures may be obtained as follows: 32° F.: Into a wooden or indurated fiber bucket put 1 gal. of water, and any convenient amount of ice, provided only sufficient is used. 20° F.: 1 gal. of water, 15 lbs. of cracked ice, and 1½ lbs. of dry common salt. 10° F.: 1 gal. of water, 15 lbs. of cracked ice, and 3 lbs. of dry common salt. Zero F.: ½ gal. of water, 15 lbs. of cracked ice, and 5 lbs. of dry common salt. A little experience will enable the temperatures desired to be reasonably well controlled. Of course, the melting of the ice continually dilutes the salt solution, and if fairly constant temperature for some time is desired, occasional small additions of salt will be needed. For temperatures below zero F., crystallized calcium chloride may be used in place of common salt. So much depends on the amounts of the materials used, and especially when dealing with such low temperatures, on the appliances, and the protection given to the vessels in which the cold is produced that it is perhaps hardly wise to try to give proportions. It may be said, however, that 3 lbs. of finely crushed ice, and 2 lbs. of crystallized calcium chloride in a properly cooled and protected vessel, will give 10° below zero F., and 3 lbs. of finely crushed ice, and 3¾ of crystallized calcium chloride under the same conditions, will give 20° below zero F. A few experiments are worth more than a good many directions, and the principles involved seem perfectly clear.

For chilling point, when it is desired to know at what temperature flocks or scales of congealed constituents of more or less opacity takes place. Use the way of obtaining the desired temperatures described in the preceding paragraph, except that in going down the scale temperatures 5° apart are produced by the proper mixtures, and used as described under "operation."

Notes and Precautions: It will be observed that this method so far as cold test is concerned, consists practically in freezing the liquid to be tested, and then taking the temperature of the flowing mass when it will just flow from one end of the bottle to the other. It is quite well known that other characteristics of the cooling of liquids have been made use of as cold test, also other methods of manipulation have been prescribed. For example, the temperature at which flocks of congealed constituents begin to separate has been called the cold test of a liquid; or, in other words, the liquid has been said to stand cold test if it remained perfectly clear at any given temperature. Still further the cold test of a liquid was formerly taken by putting an ounce or so in a bottle, as described above, hanging a thermometer in the center of it, and then exposing the whole thing to cold without agitation, and observing the temperature at which the last part of the liquid or that immediately surrounding the thermometer became congealed. In view of this diversity of practice the distinctions given in the earlier part of this method seem desirable.

It is well known that most liquids whose cold test is desired contain constituents which congeal at different temperatures; in other words, they are not homogenous. It is also well known that when liquids made up of constituents which congeal at different temperatures are exposed to the slow action of cold, the constituents of highest congealing point solidify first, next to the sides of the bottle, provided the liquid is left undisturbed, and a little later, if the source of cold is low enough, constituents of lower congealing point solidify, and so on gradually forcing to the center of the bottle those constituents which have the lowest congealing point. Furthermore, it is well known that the separation of constituents of different congealing points, as described above, is affected by the rate at which the cold penetrates the mass of liquid. If the rate is very slow, that portion of the liquid around the thermometer which congeals last will show a much lower figure for cold test than if the rate of cooling is more rapid. In view of these facts, it is evident that the method formerly in use— viz.: observing the temperature at which that part of the liquid around the thermometer, as above described, congealed—did not give the cold test of the whole liquid, but of that constituent which congealed at the lowest temperature, and that the figure obtained was a function of the rate of cooling. It is believed that the method described above obviates these difficulties, and that with some experience and sufficient care in the manipulation, duplicate results can be obtained on the same sample, which will agree within 2° or 3°.

It will be noted that the directions require stirring and mixing of the liquid, both in taking cold test and chilling points. The object of this is to enable the cold test or chilling point of the whole liquid to be obtained, rather than that of constituents of it, as explained above. Moreover, without stirring and mixing, the whole mass would not be of the same temperature, since the thawing or access or heat in taking cold test proceeds from the outside toward the center. It is, of course, recognized that the chilling point is affected by the agitation of the liquid during cooling; in other words, it is probable flocks or scales separate at a little higher temperature with agitation of the liquid than if it is allowed to remain perfectly quiet. No practical method of getting the whole of the liquid cooled to the same temperature within reasonable time is known, however, except to stir and mix, and since the manipulation is prescribed, no unfairness results. It may be queried whether the amount of stirring does not have an influence, but experiments seem to indicate that if a liquid is close to the margin, any stirring will be sufficient to cause flocks or scales to separate, while if the limit is somewhat wide, considerable stirring will not bring turbidity.

With proper appliances a very large number of cold tests can be made in a day. Also chilling points to see if the liquid remains clear at any specified temperature, require no very elaborate manipulation. Chilling points to see at what temperature turbidity begins are much slower, since they require constant watching and manipulation.

In the specifications, for the supply of oils to the various railroads, it is generally stated what degree is required for the cold test. Thus the Pennsylvania Railroad Company requires as follows:—

150° Fire Test Oil—must not become opaque or show cloud at a temperature of 32° F.

300° Fire Test Oil—must not become opaque or show cloud when the sample has been 10 minutes at a temperature of 32° F.

Paraffine and Neutral Oils—from October 1st to May 1st have a cold test above 10° F., and from May 1st to October 1st have a cold test above 32° F.

Well Oils—same as Paraffine and neutral oils.

C. B. & Q. R. R.—require cold test for Black Engine oils as follows:

"Summer oil," must flow at 60° F. or above.
"25° oil," must flow at 30° F. or above.
"15° oil," must flow at 20° F. or above.
"Zero oil," must flow at 5° F. or above.

Erie R. R.—require cold test for Petroleum Stock oil as follows:

"Summer oil," not above 60° F.
"15° oil," not above 15° F.
"Zero oil," not above 0° F.
"5° below Zero oil," not above 5° below Zero F.

300° Fire Test Oil (*Illuminating oils*)—must not become opaque or show cloud when the sample has been 10 minutes at a temperature of 0° F.

3. Viscosity

The test for viscosity or "body" of lubricating oils is an important one.

By comparison with standards an oil may be rated as to its viscosity thus giving one of the values required for a lubricant.

Engler's apparatus—probably the first used for this purpose, is made of metal (copper) and its general plan is shown in Fig. 7.

This instrument is the standard for determining the viscosity of oils in Germany, and is also a standard in this country. Recommended by U. S. Bureau of Standards for use unless another form of viscosimeter is called for in the specification.

In using this instrument the viscosity of an oil is stated in seconds required for 200 cc. of the oil to run into the flask, 240 cc. of the oil being placed in the viscosimeter. Water usually requiring from 50 to 53 seconds at 20° C.

Heat can be applied to the water-bath, the viscosity being determined at any temperature required up to 100° C. Higher temperatures to 360° C. can be secured by filling the outer vessel with paraffine instead of water.

Engler recommends that all viscosities be compared with water thus:

If water requires fifty-two seconds for delivery of 200 cc. into

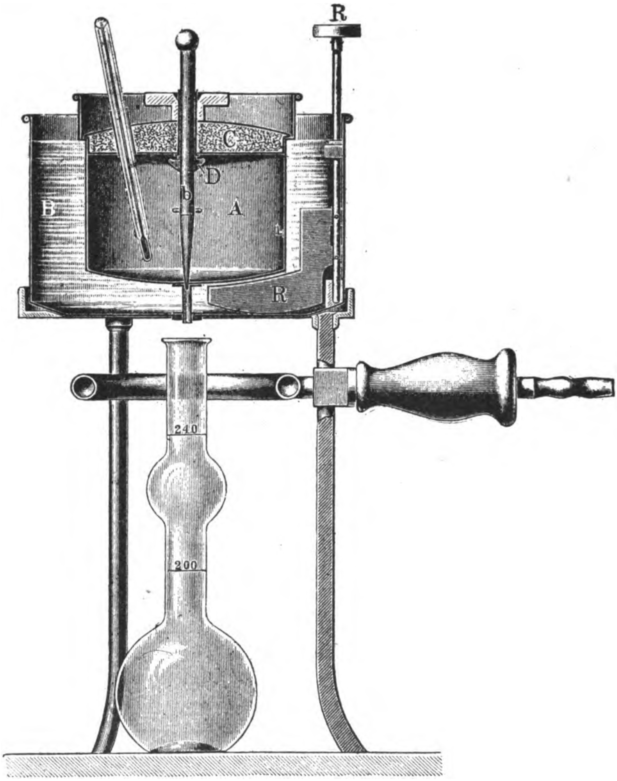

Fig. 7.—Engler Viscosimeter.

the receiving flask, and the same amount of an oil under examination requires 130 seconds, the ratio is determined by $\frac{132}{52} = 2.50$, the oil thus having a viscosity of 2.5 times that of water.

"The American Society for Testing Materials," Report of committee D-2, state as follows:

In case it is desired to correct for specific gravity of the oil, the following formula which gives the results in specific viscosity can be used:

$$\text{Sp. viscosity} = \text{sp. grav.} \times \frac{\text{time of efflux of oil}}{\text{time of efflux of water}} \times 7.32.$$

If it is necessary to use a quantity of oil less than 240 cc. the following quantities can be employed and multiplied by the corresponding factor:[1]

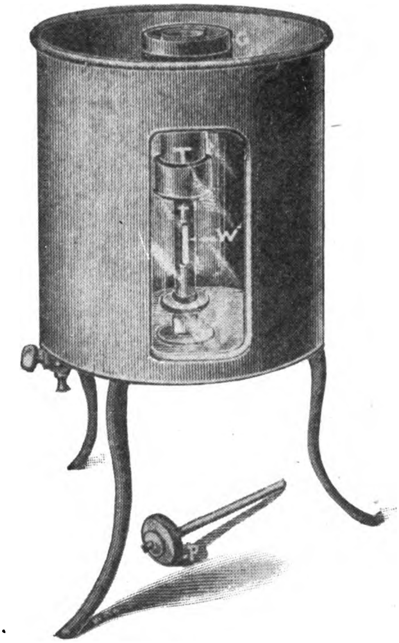

Fig. 8.

Amount of oil put in, cc.	45	50	60	120
Amount of oil run out, cc.	25	40	50	100
Factor to change to 200 cc. run out and 240 cc. put in	5.55	3.62	2.79	1.65

The Saybolt Viscosimeter (Fig. 8), is used by the chemists of the Standard Oil Co., and is one of the standards in this country. It is made of metal and glass.[1]

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[1] Gans, *Chemische Revue der Fette und Harz-Industrie*, Vol. VI, p. 221.

Its dimensions are as follows:

Diameter of overflow filling gage cup..............	51.0 mm.
Depth of overflow filling gage cup.................	13.0 mm.
Diameter of main cylindrical tube..................	30.0 mm.
Depth from starting head to outlet jet	113.0 mm.
Length of outlet jet	13.0 mm.
Diameter of outlet jet.............................	1.8 mm.
Charging quantity..................................	70.0 cc.

The specific viscosity obtained with this instrument is not the same as with the Engler: tables for the conversion of the readings of one instrument into those of the other are in preparation by the United States Bureau of Standards. Meanwhile the comparison by Saybolt can be used, and is given in Table I.

TABLE I.—COMPARISON OF VISCOSITY AS DETERMINED BY THE ENGLER AND SAYBOLT VISCOSIMETERS.

Sample No.	Engler. 200 cc. of oil at 68° F.		Engler. 200 cc. of oil at 100° F.		Saybolt universal 60 cc. of oil at 100° F.		Greater viscosity by Saybolt, per cent.
	Time of efflux, seconds	Viscosity[1]	Time of efflux, seconds	Viscosity[2]	Time of efflux, seconds	Viscosity[3]	
312......	106	2.00	79	1.49	49	1.66	11
304......	304	5.73	146	2.75	101	3.36	29
302......	666	12.56	260	4.90	182	6.06	23
317......	852	16.06	319	6.02	223	7.43	23
318......	1,230	23.20	437	8.24	305	10.16	23
306......	1,300	24.53	451	8.51	313	10.43	22
309.....	1,765	33.30	574	10.83	399	13.30	22
310......	2,120	40 00	688	12.98	481	16.03	23
Average	—	—	--	11.49	- -	14.37	

The points in favor of the Saybolt instrument are:

1. The large ratio of the volume of the bath of water to the volume of oil tested. This is important in keeping the test sample at the desired temperature during the test.

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[1] Full description will be found in "Oil Analysis," by A. H. Gill, pp. 24-26, and *Journal Anal. Chem.*, V, *pp.* 324-353, *Stillman.*

[2] Compared with 200 cc. of water at 20° C., 53 seconds.

[3] Compared with 60 cc. of water at 100° F., 30 seconds.

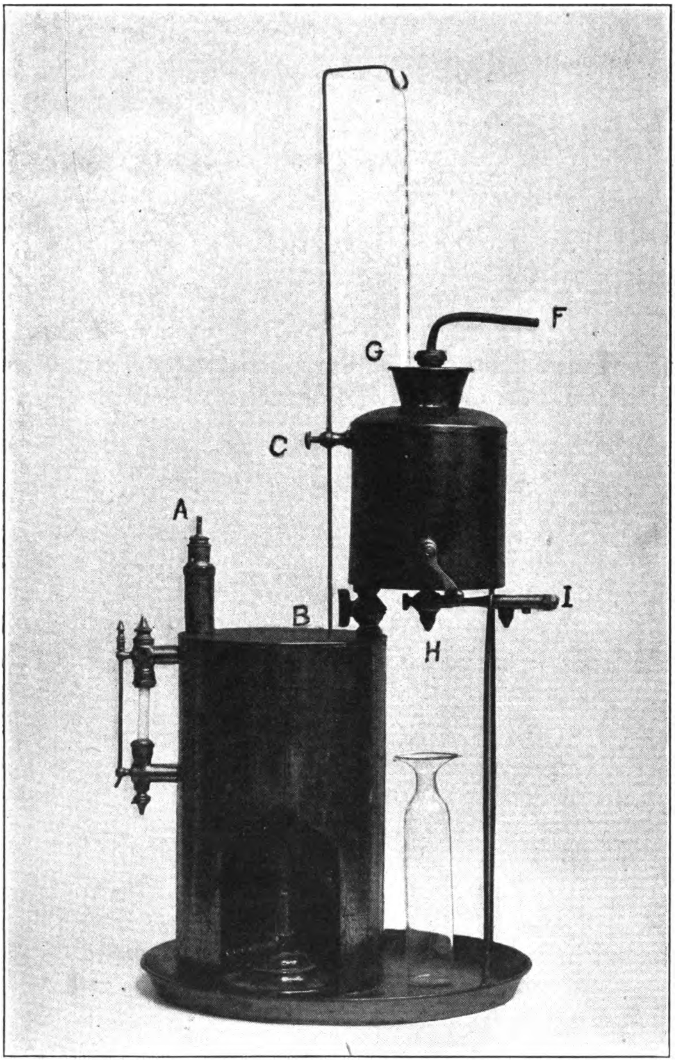

Fig. 9.—Tagliabue's Improved Viscosimeter.

2. The ready means of adjusting the quantity of oil with which the instrument is filled.

3. The simple device for stopping the jet, permitting of no injury to it.

4. The heating device which excludes the danger of superheating the outlet jet.

5. The fact that it uses a smaller quantity of oil, and requires less time than any other viscosimeter.

Tagliabue's Viscosimeter (Fig. 9), is used to a very large extent by the manufacturers of lubricating oils in the United States. The following are the directions for its use:

TO TEST THE VISCOSITY OF OILS AT 212° FAHRENHEIT

Pour water into boiler through opening A, after unscrewing safety valve until water gauge shows that the boiler is full. See that stop-cock B is open, making direct connection between boiler and upper vessel which surrounds the receptacle in which the oil to be tested is placed: Place wire holder in set nut C and suspend thermometer so that the bulb of thermometer will be about one quarter of an inch from the bottom of oil bath. Then after carefully *straining* 80 cc. of the oil to be tested, which of course must be warmed in the case of very heavy oils, pour same into oil bath. Close stop-cocks D and E. Screw the extension F with rubber hose attached into coupling G and let the open end of hose be immersed in a vessel of water which will prevent too large a loss of steam. Place lamp or Bunsen burner under boiler; screw steel nipple marked 212 on to stop-cock H; the apparatus is now ready to use. After steam is generated wait until thermometer in oil bath shows a temperature of 210½ or 211°; then place the 60 cc. test-glass under stop-cock H so that the stream of oil strikes the side of test-glass, thereby preventing forming of air bubbles; and when the thermometer indicates its highest point open the faucet H simultaneously with the starting of the watch which is supplied with each instrument.

When the running oil reaches the 60 cc. mark in the neck of the test-glass the watch is instantly stopped, and the number of seconds noted. Then multiply the number of seconds by two, and the result will be the viscosity of the oil. For example: If 60 cc. of oil runs through in 101½ seconds the viscosity would be 203,

It is best to repeat the test until sufficient skill is attained by practice for uniform results.

It is also necessary to keep the oil well stirred before making test in order to have the oil at a uniform temperature.

TO TEST THE VISCOSITY OF OILS AT 70° FAHRENHEIT

Screw the steel nipple marked 70° on the faucet H, close stop-cock B, thereby closing communication between boiler and upper vessel. Also close stop-cock E. Fill upper vessel, through opening G, with water, as near a temperature of 70° as possible, also having the oil to be tested, at the same temperature. Hang the thermometer in position, and after stirring the oil thoroughly, blow through the rubber tube at D, thoroughly mixing the water. Should the thermometer show a lower or higher temperature than 70°, add cold or warm water until the desired temperature is obtained. Then measure carefully 90 cc. of oil to be tested, placing it in the machine, and when everything is ready, open stop-cock, and start watch at the same time, and allow 70 cc. to pass through the nipple, and as soon as the test-tube is filled to the 70 cc. marked in the neck, turn off the stop-cock and stop watch at the same moment. Should it take the 70 cc. 96 seconds to run through the nipple, multiply this by two, and you will have the viscosity of the oil, which is 192 seconds. (This multiplication by 2 is to render readings comparable with the Saybolt Viscosimeter).

Riehle'-Stillman Viscosimeter

It is important often also that the portion of the viscosimeter containing the oil to be tested should be small, holding not over

100 cc. of oil. Samples of oil are generally sent out in the oil trade in long 4-ounce vials, the latter technically called "oils."

In nearly all viscosimeters more than 150 cc. of oil are required for the viscosity test and unless larger samples have been sent for examination, the viscosity test is dependent upon another and larger sample. If, however, the viscosimeter holds only 100 cc. of oil this test can be made first of all. That is to say, suppose a 4-ounce sample of oil is sent to a chemist to determine flash test, fire test, cold test, viscosity at 15° C. and 100° C., percentages of animal or vegetable oil, and mineral oil: First determine the viscosity of 50 cc. of the oil at the two temperatures desired; this in no way effects the composition of the oil or interferes with using a portion of the 50 cc. for flash test, fire test, or freezing test. In this manner 100 cc. would be sufficient for the above examinations of the oil.

The viscosimeter which accomplishes this end, and is used in the laboratories of the Stevens Institute of Technology, is thus briefly described: The inner compartment to contain the oil to be tested is circular, 2½ inches in diameter at the top, 6½ inches long, converging at the base to the outlet $^1/_{64}$ inch in diameter (Fig. 10). This outlet is closed by a rack and pinion shutter, which works with great rapidity with the handle, shown in the drawing at the side of the viscosimeter. The outer vessel (to contain water, or, if a temperature above 100° C. is desired, melted paraffine) is circular, 5 inches in diameter and 7 inches in depth. Underneath the copper bottom of the viscosimeter is a coiled iron pipe with gas-jet openings. This can be regulated to obtain the necessary heat. At the top of the inner vessel is a copper cover containing the thermometer. The mercury bulb of the latter should reach about 3 inches in the oil.

The process of determining the viscosity of an oil is similar to the method of Engler; *viz.,* 100 cc. of the oil are placed in the inner vessel, and heat applied to the viscosimeter (the outer vessel being filled with water) until the required temperature is reached and the oil maintained at that heat for five minutes before allowing it to run into the 50 cc. graduated flask.

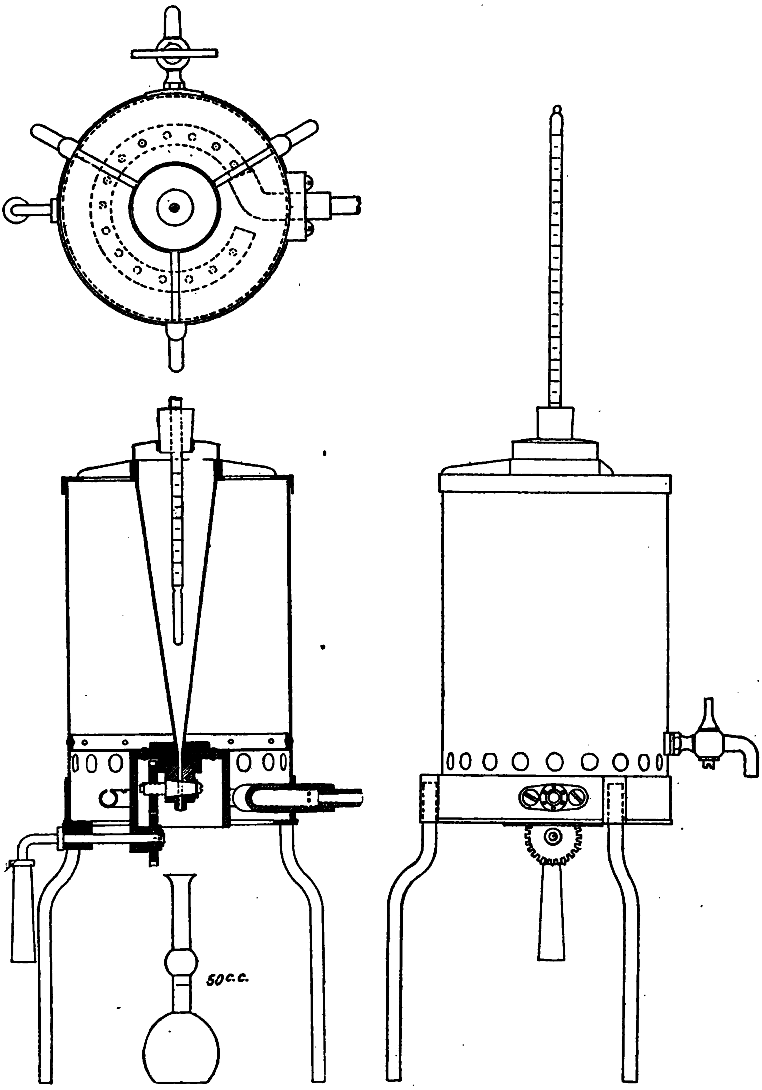

Fig. 10.
(Manufactured by Riehlé Bros. Testing Machine Co., Phila., Pa.)

When the graduation mark in the neck of the flask is reached by the oil, the cut-off valve is turned, shutting off the supply of oil. The time required for 50 cc. of oil to enter the flask from the viscosimeter, in seconds, is called the viscosity. It is becoming customary now to compare the viscosity of oil with water as

50 cc. taken for viscosity.	Seconds at 20° C. = 68° F.	Seconds at 50° C. = 122° F.	Seconds at 100°C. = 212° F.	Seconds at 150°F. = 302° F.	Seconds at 200°C. = 392° F.
Water	30	..	..	..	..
Prime lard oil	110	58	38	32	..
No. 1 lard oil	140	60	36	32	..
XXX lard oil	146	62	36	32	..
Prime neat's foot oil	120	56	36	32	..
White neat's foot oil	140	56	36	32	..
Gelatine oil	solid	solid	solid	720	70
Rosin oil, 1st run	..	..	38	30	..
Rosin oil, 2d run	140	46	30	28	..
Rosin oil, 3d run	150	44	30	28	..
Dog-fish oil (Pacific)	100	52	34	32	..
Right whale oil (Pacific)	116	54	36	30	30
Natural bow head oil (Pacific)	94	54	36	32	..
Bleached whale oil	104	54	36	32	..
Natural winter sperm oil (Pacific)	66	44	32	30	..
Bleached sperm oil (Pacific)	58	44	32	30	..
Natural spring sperm oil	60	44	34	32	30
Bleached spring sperm oil	64	44	32	30	..
Natural winter whale oil (Atlantic)	106	52	34	32	..
Bleached winter whale oil (Atlantic)	86	52	36	30	..
Extra bleached winter whale oil (Atlantic)	111	56	36	32	..
Natural spring whale oil (Atlantic)	114	52	36	32	..
Bleached spring whale oil	104	52	36	32	..
Porpoise head oil	68	60	32	30	..
Sea elephant oil	102	72	34	32	.
Bank oil	78	60	34	32	..
Prime crude menhaden oil	78	48	34	32	..
Brown strained menhaden oil	84	48	36	32	..
Light strained menhaden oil	80	48	34	32	..
Natural winter menhaden oil	82	50	34	32	..
Bleached winter menhaden oil	68	48	34	32	..
Extra bleach'd winter white menhaden oil	78	48	34	32	..
Castor oil	146	190	46	34	30
"White Seal" castor blown oil	..	360	56	40	40
Prime qual. summer white cotton-seed oil	102	52	34	30	..
Prime qual. winter white cotton-seed oil	114	54	36	30	..
Rape oil	151	52	40	32	30
Olive oil	126	48	36	32	30

the standard; thus, suppose 50 cc. of a mineral oil required seventy seconds, at 60° F., and the same amount of water required sixteen seconds, at 60° F., the ratio of viscosity would be $^{70}/_{16}$, or 4.3 times that of water. The preceding determinations of viscosity were made by this instrument.

"The Doolittle Torsion Viscosimeter"[1]

used in the railroad laboratories of the Philadelphia and Reading Railroad Company, is briefly described as follows:

A steel wire is suspended from a firm support and fastened to a stem which passes through a graduated horizontal disk, thus measuring accurately the torsion of the wire. The disk is adjusted so that the index point reads exactly zero, thus showing that there is no torsion in the wire (Fig. 11).

A cylinder two inches long by one and a half inches in diameter, having a slender stem by which to suspend it, is then immersed in the oil and fastened by a thumb-screw on the lower part of the stem to the disk. The oil is surrounded by a bath of water or paraffine wax according to the temperature at which it is desired to take the viscosity. This temperature being obtained while the disk is resting on its supports, the wire is twisted 360° by means of the knob at the top. The disk being released, the cylinder rotates in the oil by virtue of the torsion of the wire.

The action now observed is identical with that of the pendulum.

If there was no resistance to be overcome, the disk would revolve back to zero, and the momentum thus acquired would carry it to 360° in the opposite direction. What we find is that the resistance of the oil to the rotation of the cylinder causes the revolution to fall short of 360°, and that the greater the viscosity of the oil the greater will be the resistance and hence the retardation. We find this retardation to be a very delicate measure of the viscosity of an oil.

There are a number of ways in which this viscosity may be expressed, but the simplest is found to be directly in the number of degrees of retardation between the first and second complete arcs covered by the pendulum. For example, suppose we twist the wire 360° and release the disk so that rotation begins. In order to obtain an absolute reading to start from, which shall be independent of any slight error in adjustment, we ignore the fact that we have started from 360°, and take as our first reading the end of the first swing. Suppose our readings are as follows:

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[1] *J. Am. Chem. Soc.*, 15, 173.

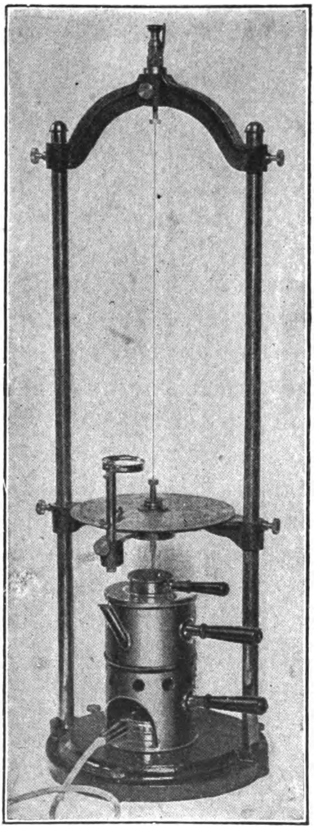

Fig. 11.—Doolittle Viscosimeter.

Right, 350; left, 338; right, 328; and keeping in mind the vibrations of the simple pendulum we will see at once that we

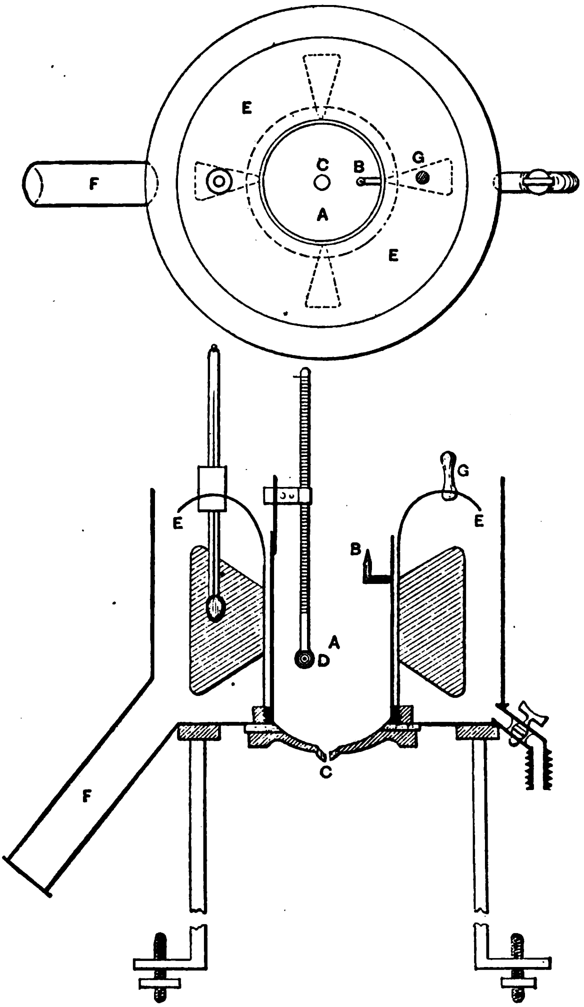

Fig. 12.—Redwoods's Viscosimeter.

have read two complete arcs whose difference is 22° computed as follows:

1st arc, Right 350° + Left 338° = 688°
2d arc, Left 338° + Right 328° = 666°
22° retardation.

In order to secure freedom from error we take two tests—one by rotating the wire to the right, and the second to the left. If the instrument is in exact adjustment these two results will be the same, but if it is slightly out, the mean of the two readings will be the correct reading.

It will also be noticed that if the exact retardation due to the oil alone is to be obtained we must subtract the factor for the resistance due to the air and the wire itself. These are readily obtained by allowing the cylinder to rotate in the air and determining the retardation exactly as we have done above. This factor remains constant for each instrument and is simply deducted from all results obtained.

The Redwood[1] Viscosimeter (Fig. 12), is on the general principle of the Engler. This is the standard viscosimeter for the English oil trade.

The Scott Viscosimeter

This instrument is used largely in the oil industry and also for taking the viscosity of glue solutions, etc. It is a modified Engler.—Figs. 13 and 14.

It consists of a metal cup with an opening at the bottom, which is kept closed by a ball valve; the inner oil cup rests in an outer casing, which is kept filled with water.

To make a test, first fill the oil cup with the oil to be tested, fill the outer casing or jacket with water, place an accurate thermometer in the oil and bring the temperature to the required point by cooling or heating the water.

Place a 50 cc. flask under the oil discharge, and with a stop watch ready, start the oil to flowing by pressing down on the lever which raises the outlet valve.

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[1] *J. Soc. Chem. Ind.*, 5, 158.

When the oil has reached the 50 cc. mark on the flask, stop the watch. The number of seconds required for the 50 cc. to flow out is noted.

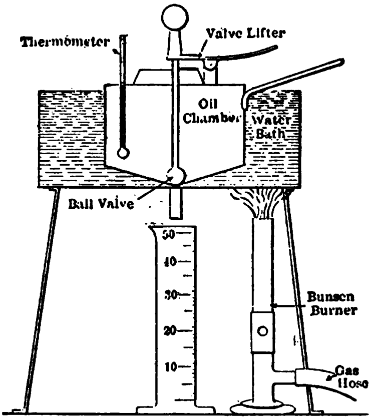

Fig. 13.

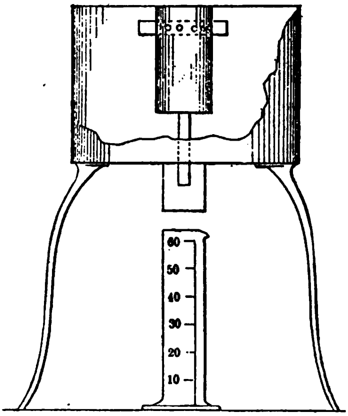

Fig. 14.

Comparison of Viscosities of Saybolt's "A" Viscosimeter and Doolittle's Viscosimeter.

(A. H. Gill)[1]

Oil.	Spec. Grav.	Saybolt. Seconds.	Doolittle. Grams of sugar.
Stove gasoline	0.680	23.	0.0
Water	1.000	25.	0.0
Kerosene, 150° Fire test	0.783	26.8	3.3
Lantern, 300°	0.827	36.4	49.8
Spindle	0.849	57.9	62.8
Spindle	0.851	60.0	65.5
Sperm	0.880	102.0	73.5
Spindle	0.863	115.0	74.3
Spindle	0.868	156.0	78.4
Loom	0.888	203.0	80.6
Lard	0.916	215.0	82.8
Mineral	0.907	224.0	83.5
Neat's-foot	0.916	250.0	83.7
25° B. Paraffine	0.900	272.0	84.2
Mineral	0.903	339.0	86.2

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[1] Oil Analysis, p. 158.

4. Iodine Absorption

The determination of the iodine absorption of an oil is probably the most important chemical test for recognition quantitatively in a mixture of animal or vegetable oils with mineral oils. Introduced by Hubl[1] it has since maintained this position, though other chemists have introduced the bromine absorption and others of similar character. They have not been adopted with the confidence of the iodine process.

Warren[2] draws attention to the fact that Chateau in his "Essais Perosnnelles," p. 70, used the iodine absorption in a manner similar to Hubl many years previously.

In a mixture of two fatty oils with a mineral oil, the best results are obtained by saponifying and separating the fatty acids from the mineral oil. The iodine absorption of the mixed fatty acids is then taken, and where the nature of them has already been shown by color tests, etc., their proportion can be indicated by the following formula:

$$x = \frac{100\,(I - n}{m - n}.$$

Where x = the percentage of one fat,
y = the percentage of the other,
I = iodine degree of mixture,
m = iodine degree of fat x,
n = iodine degree of fat, y.

The method is as follows:[3]

Twenty-five grams of iodine and 30 grams of mercuric chloride are each dissolved in 500 cc. of 95 per cent. alcohol, uniting the two solutions, and allowing to stand several hours before use.

It is then standardized by tenth-normal thiosulphate sodium solution. The process of the determination of the iodine absorption of an oil is as follows: 0.1 to 0.5 gram of the fat or oil is dissolved in 10 cc. of purest chloroform in a well-stoppered flask, and 20 cc. of the iodine solution added. The amount must be finally regulated so that after not less than two hours' digestion the mixture possesses a dark brown tint; under any circumstances it is necessary to have a considerable excess of iodine (at least double the amount absorbed ought to be present), and the digestion should be from six to eight hours. Some potassium iodide solution is then added, and the whole diluted with 150 cc. of water, and tenth-normal thiosulphate solution delivered in till the color is nearly discharged. Starch is then added, and the titration finished in the usual way.

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[1] *Ding. poly. J.*, 253, 281.

[2] *Chem. News*, 26, 188.

[3] Sutton's "Volumetric Analysis," p. 343.

If more than two fatty oils are present in a mixture with mineral oil, the method of Warren[1] can be used.

The following determinations of the iodine absorption made in my laboratory are indicative of the variations of the absorption by the different oils:

Prime lard oil	76.4	77.2
No. 1 lard oil	69.8	69.9
XXX lard oil	65.1	65.6
Oleo oil	51.6	51.6
Prime neat's foot oil	80.1	82.0
Horse oil	82.3	82.5
Natural bow-head whale oil	130.5	131.1
Natural winter whale oil	121.1	126.0
Extra bleached winter white oil	124.9	126.1
Bleached spring winter white oil	126.1	126.2
Crude sperm oil	82.3	82.3
Prime quality winter white cotton-seed oil	114.2	114.9
Prime quality summer white cotton-seed oil	110.2	110.6
Prime quality winter yellow cotton-seed oil	115.9	118.6
Prime quality summer yellow cotton-seed oil	104.0	104.4
Olive oil	81.0	83.0
Herring oil	122.1	123.8
Dog-fish oil	102.7	104.7
Porpoise head oil	28.9	29.1
Rosin oil, second run	92.1	93.4
Rosin oil, third run	90.4	92.2
Rape oil	94.0	106.8
Corn oil	111.0	115.0

5. Flash and Fire Test

The flash point is the degree of temperature at which ignitable volatile vapors are given off by the oil, producing a flash when brought in contact with a small flame. The fire test is a continuation of the flash test until the oil permanently ignites.

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[1] *Chem. News*, 62, 215; *J. Anal. Appl. Chem.*, 5, 215.

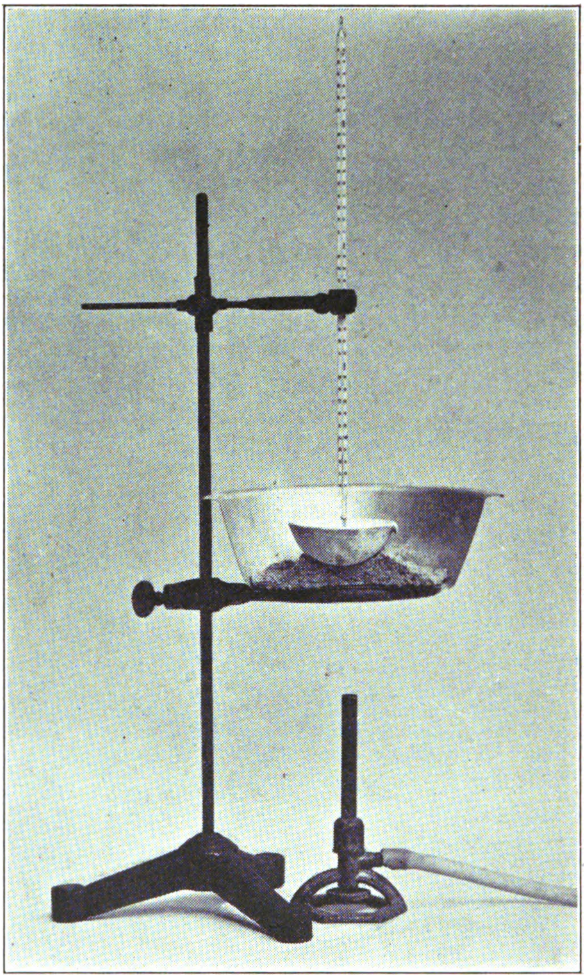

Fig. 15.—Apparatus for Determining the Flashing and Burning Points of Combustible Liquids.

The method used by the chemists of the P. R. R. Co. is as follows:

Operation

Have ready the apparatus shown in the cut. Fig. 15. Light the lamp, put under the center of the dish, adjust it as previous experience has indicated is necessary to secure the proper rate of heating, and allow the apparatus to get warm. Fill the porcelain dish to within about one-fourth of an inch of the top with the liquid to be tested, adjust the thermometer in the centre of the liquid, and so as to reach nearly to the bottom of it, but not quite touching the dish. Adjust the flame so that the temperature of the liquid in the dish rises at the rate required for the liquid being tested, and when the temperature reaches a desired point, apply the test flame by passing it slowly, entirely across the dish, about a half an inch above the level of the liquid and just in front of the thermometer. Allow the liquid to rise in temperature until another testing point is reached and then apply the test flame again in the same manner. Proceed in this way until the vapor from the liquid above it ignites with a slight flash. The temperature shown by the thermometer when this is the case is the flashing point of the liquid. Continue the heating and testing in the same manner until a point is reached where the liquid takes fire. The reading of the thermometer when this is the case is the burning point of the liquid. Remove the thermometer, blow out the flame, or smother it by sliding a watch glass over the top of the porcelain dish, empty the dish and wipe it out, when the apparatus is ready for another test.

Apparatus

The principal portion of the apparatus required in determining the flashing and burning points is shown in the cut (Fig. 15). It consists, as is seen, of an iron tripod, fitted with a rod and clamp to sustain the thermometer, and supporting a small pan or dish partly filled with sand. On the sand and partly bedded in it is a royal Berlin porcelain dish about 2¾ in. in diameter and 1 in. deep, to hold the liquid to be tested. Underneath the iron dish is a Bunsen burner to furnish heat. The thermometer may be any good chemical thermometer, but those with milk or paper scale, and graduated to single degrees, are easiest read.

The testing flame may be obtained in a variety of ways. A fine string or coarse thread was used for many years. This, when lighted, gives a bead of flame as large as a small pea. A glass tube drawn out to rather a fine jet and connected by a rubber hose with a gas supply is much better. The flow of gas can readily be adjusted to give a jet about 1/4 in. long and 1/8 in. in diameter, which is abundant in size. The glass tube fuses up sooner or later, and therefore best of all is a porcelain tube, such as is used with crucibles for ignition in hydrogen, attached by a rubber tube to a gas supply. The flame should be of the size given above.

NOTES AND PRECAUTIONS

The philosophy of the method of taking flashing and burning points of combustible liquids is simple. All liquids when heated to sufficient temperature give off vapors, the vapor being either a part of the liquid itself or the products of decomposition from it. Moreover, most liquids whose flashing and burning points are desired, notably the petroleum products, contain some constituents which are volatile at lower temperatures and some which are volatile at higher temperatures, so that when heated by a constantly increasing temperature, vapors pass off in constantly greater amount. The process being conducted in the air, the vapors passing off from the liquid obviously mix with the air above it, and in course of time, if the heating is properly managed, a slightly explosive mixture of vapor and air exists just above the liquid. If now, a flame is passed through this explosive mixture it is ignited, and a more or less violent explosion, or "flash," as it is called, follows. The lowest temperature at which enough vapor escapes from the liquid to form this explosive mixture is called the flashing point of the liquid. In like manner, by continuing the heating and raising the temperature higher and higher, the amount of vapor issuing from the surface of the liquid becomes continually greater and greater, and the explosions become more and more frequent, if the test flame is applied often enough, until finally the amount of vapor above the liquid becomes so great that it will take fire and burn continuously. The lowest temperature at which this takes place is called the burning point of the liquid. It is obvious, therefore, that any conditions which have an influence on the generation of vapor from combustible liquids, or which have an influence on the mixing of this vapor with the surrounding air, or which influences its dissipation from the surface of the liquid, will have an influence on the flashing and burning points of the liquids. It is also obvious that the apparatus used and the method or manipulation will have an influence on the figures obtained, so that the flashing and burning points of any given sample of combustible liquid, obtained by means of one apparatus and method of manipulation, will not necessarily be the same as that obtained by means of a different apparatus and a different method of manipulation, even on the same sample of liquid.

The condition which has the most influence on the flashing and burning points of combustible liquids is the rate of heating the liquid, and this is at the same time the most difficult condition to control. The more rapid the rate of heating, the lower the flashing and burning points, and in view of the principles given above, it is easy to see why this should be so. Obviously a liquid could be heated so slowly that the vapor formed would for a long time pass off into the air by diffusion, and the amount necessary to indicate the flashing and burning points be obtained only with much higher temperatures. On the other hand, a liquid can be heated so rapidly that the flashing and burning points are practically the same, and these many degrees below the figures obtained by slower heating. The rate of heating used in the Pennsylvania Railroad Laboratory is as follows:

For 150° fire-test petroleum,	12° F. per minute.
For 300° fire-test petroleum,	15° F. per minute.
For Paraffiine oil,	15° F. per minute.
For well oil,	15° F. per minute.
For 500° fire-test petroleum,	15° F. per minute.
For all other combustible liquids of high burning point,	15° F. per minute.

The adjustment of the rate of heating can best be learned by experience and constant watching of the thermometer. If the amount of sand in the dish is moderately large, and the temperature of the room constant, and the place where the tests are made free from drafts, the flame can be adjusted after some experience so as to give a close approximation to the required rise in temperature. But the thermometer must, of course, be constantly watched, and it is many times necessary to repeat a test even two or three times with readjustment of the flame on account of failure to secure the proper rate of heating. Moreover, each time the liquid is flashed a small amount of heat is generated, which affects the thermometer, and if the flashing is done very frequently this may be a very serious cause of disturbance of the rate of heating.

The lower the flashing and burning points of the liquid being tested, the more difficult the manipulation to secure the required rate of heating.

In view of the difficulties introduced by frequent flashing, it is advisable to limit the number of times the test flame is applied as much as possible, and accordingly in the Pennsylvania Railroad Laboratory the test-flame is first applied as follows:

For 150° fire-test petroleum,	at 123° F.
For 300° fire-test petroleum,	at 242° F.
For paraffine oil,	at 291° F.
For well oil,	at 242° F.
For 500° fire test petroleum,	at 487° F.
For wood preservative,	at 165° F.

In all cases after the first the test-flame is applied after each 7° rise in temperature, until the burning point is reached.

The method of applying the test-flame has an important influence. By skillfully and adroitly passing the test-flame down alongside the thermometer to within ¼ in. of the liquid and quickly pulling it out again, considerable higher figures may be obtained for the flashing and burning points than with the manipulation specified. Apparently near the center of the dish, alongside the thermometer, the air and vapor are not as well mixed, or at least are not in condition to ignite as readily as at the side of the dish. By the prescribed manipulation the chances for juggling with the results are diminished, since the test-flame passes twice through that part of the mixture of vapor and air which is most readily ignited—namely, the part at the edges of the dish.

It is impossible to make satisfactory tests for flashing and burning points in a place subject to drafts.

Among the variables affecting the flashing and burning points of combustible liquids, which are made constants by the apparatus specified above, may be mentioned the amount of surface exposed, the depth of liquid in the dish, and the total amount of liquid. Any or all of these being different, the same figures would not be expected, even though the manipulation was in every respect as specified. Moreover, a difference of an inch or more in the barometric pressure, which might readily occur in different parts of the country, would also have an influence on the figures obtained, the lower barometer giving the lower figures.

It is well known that thermometers change with use, especially if they are not well seasoned before going into service. It is essential, therefore, to occasionally compare with a standard, or to use new thermometers.

Occasionally an anomalous low fire-test petroleum product will be found. The flashing and burning points are very close together, whatever the rate of heating. Apparently the liquid is very closely fractionated, or is made up of constituents differing very slightly in vaporizing point, and consequently, its behavior does not conform to that characteristic of liquids having a wider range of constituents.

It is fair to say that open-cup testing of combustible liquids, even when all possible precautions are taken, is subject to some uncertainties. Duplicate tests with the same liquids, the same apparatus, and the same manipulator may disagree one point, or 7° and an attempt to get duplicate figures, representing the flashing and burning points of a liquid as close as 1° or 2° F., would probably result in failure.

All open cup testers give higher readings for the flash test than closed testers and it is generally conceded that the closed testers admit of more accurate determinations.

The Abel closed tester (Figs. 16 and 17) has been adopted by the English government, and in a modified form (Pensky-Martens) by the German government as the official instrument for this purpose.

The specifications for this instrument require that the oil cup be a cylindrical vessel, 2 inches in diameter, 2.2 high (internal), with outward projecting rim 0.5 wide, ⅜ inch from the top, and 1⅞ inches from the bottom of the cup. It is made of gun metal or brass (17 B.W.G.) tinned inside. A bracket, consisting of a short stout piece of wire, bent upward, and terminating in a point, is fixed to the inside of the cup to serve as a gauge. The distance of the point from the bottom of the cup is 0.5 inch. The cup is provided with a close-fitting, overlapping cover, made of

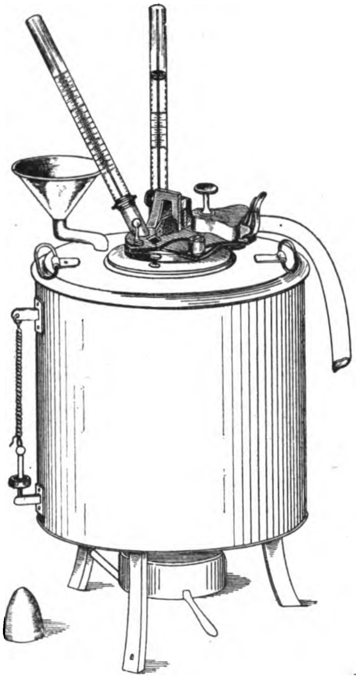

Fig. 16.

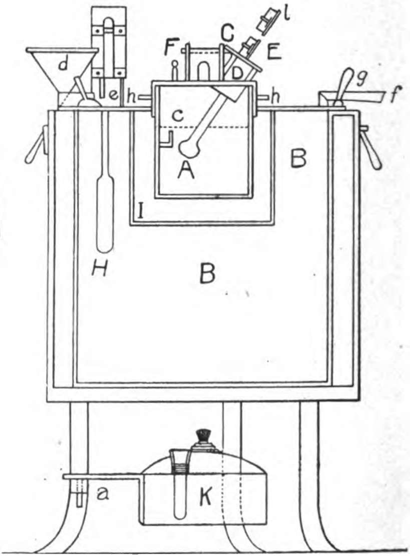

Fig. 17.

brass (22 B.W.G.) which carries the thermometer and test-lamp. The latter is suspended from two supports from the side by means of trunnions, upon which it may be made to oscillate; it is provided with a spout, the mouth of which is $^1/_{16}$ of an inch in diameter. The socket which is to hold the thermometer is fixed at such an angle, and its length is so adjusted, that the bulb of the thermometer, when inserted to full depth, shall be 1.5 inches below the center of the lid. The cover is provided with three square holes, one in the center, 0.5 inch by 0.4 inch, and two smaller ones, 0.3 inch by 0.2 inch, close to the sides and opposite to each other. These three holes may be closed and uncovered by means of a slide moving in grooves and having perforations corresponding to those on the lid. In moving the slide so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide and tilted in such a way as to bring the end of the spout just below the surface of the lid. When the slide is pushed back so as to cover the holes, the lamp returns to its original position.

The flash test of this apparatus is about 27° F. lower than the open cup apparatus, so that 73° F. Abel test is equivalent to 100° F. test, open-cup test.

Pensky-Martens Test.—Where great accuracy is required the Pensky-Martens tester should be employed. The method of operating is as follows:

Referring to Fig. 18, *E* is the oil container, which is placed in a metal heating vessel *H*, provided with a mantle *L* in order to protect the heating vessel from loss of heat by radiation. The oil-cup *E* is closed by a tightly-fitting lid (shown in plan). Through the center of the lid passes a shaft carrying the stirring arrangement, which is worked by means of the handle *J*. In another opening of the cover is fixed a thermometer. The lid is perforated with several orifices, which are left open or covered, as the case may be, by a sliding cover. This can be rotated by turning the vertical spindle by means of the milled head *G*. By turning *G*, an opening of the slide can be made to coincide with an orifice in the cover, and simultaneously a very small flame, burning at the movable jet *E*, is tilted on to the surface of the oil.

The test is performed by filling the oil into the oil-cup up to a certain mark, fixing the cover, and heating the oil somewhat rapidly at first, until its temperature is about 30° C.

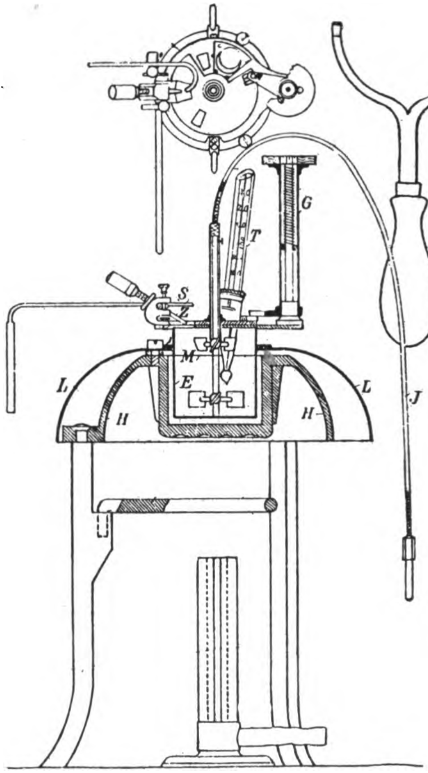

Fig. 18.—Pensky-Martens Tester.

below the expected flash point. The temperature is then allowed to rise very slowly only, by making suitable use of the wire gauze shown in the figures, so that the rise of temperature within half a minute does not exceed about 2° C. From time to time the milled head *G* is turned and the flame tilted into the oil-cup. The temperature at which a slight explosion is produced is noted as the flash point of the oil.

Treumann Apparatus.

The apparatus of Treumann (Figs. 19 and 20) is used by the chemists of the Prussian railways for the determination of the flash and fire-test of both illuminating and lubricating oils.

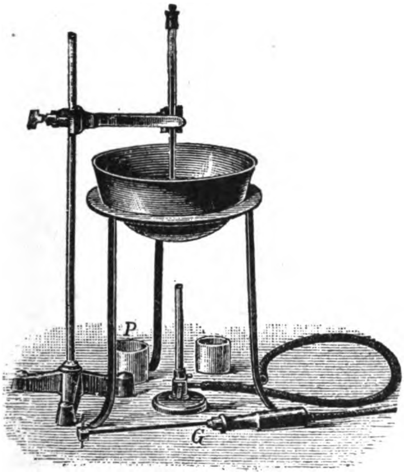

Fig. 19.

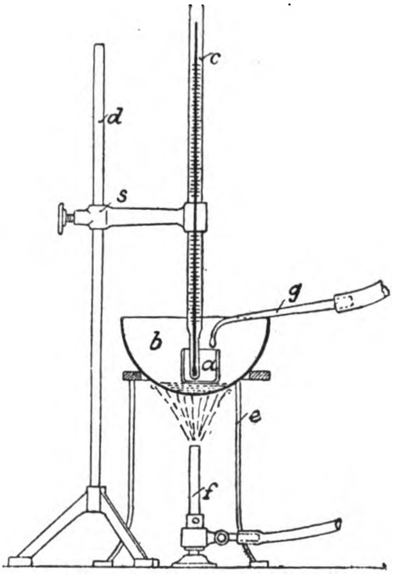

Fig. 20.

It is very similar in construction and operation to the Cleveland cup, in use in this country for the same purpose.

The larger containing vessel is of iron and contains sufficient sand to raise the bottom of the crucible containing the oil, one-half inch from the point of contact of the flame.

The flash and fire tests are required of all lubricating oils as a test of their power to resist combustion by overheating in work. Valve oils with mineral stock are especially liable to have low flash points caused by imperfect distillation in their manufacture. They should be free from any of the lighter oils (naphtha, kerosene, etc.) and should not flash under 300° F. For cylinder oils the requirement is much higher. Animal and vegetable oils used in lubrication rarely flash under 400° F.

6. Acidity

Acidity in oils is generally due to a partial decomposition of the oil with liberation of fatty acids. These latter act as corrosive agents, attacking the metal bearings of machinery, forming "metallic soaps" and producing gumming and thickening of the lubricant.

Properly refined mineral oils are free from acidity, but nearly all animal and vegetable oils possess it more or less.

In palm oil, for instance, the free fatty acids vary from 12 to 80 per cent. In 89 samples of olive oil intended for lubricating purposes, D. Archbutt[1] found from 2.2 to 25.1 per cent. of free acid (oleic), the mean being 8.05 per cent.

The action of free acid on journals, bearing, etc., as a corrosive agent, has led many engineers to include a test of free acid direct upon copper and iron.

This is done by suspending weighed pieces of sheet copper and iron in the different oils, for a number of days, heating if necessary, and determining the amount of metal dissolved by the oils.

While this test may be indicative of the acidity of oils, no ratio exists between the action upon copper and iron or even between the oils themselves in this respect, owing to the varying quantity of acid in the same oils.

Oleic acid cannot be present as a constituent of a pure mineral oil; still the acid test should be made, since poorly refined mineral oils are liable to contain small amounts of sulphuric acid left in the process of refining. The sulphuric acid is easily indicated by warming some of the oil with distilled water, adding a few drops of hydrochloric acid (dilute) and solution of barium chloride. A white cloud of precipitate shows the presence of sulphuric acid.

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[1] *Analyst*, 9, 171.

The following is the method for determining the acidity of oils as used in many of the railroad laboratories:

MATERIALS REQUIRED

½ dozen 4 ounce sample bottles.

3 10 cubic centimeter pipettes, or if desired a balance weighing milligrams.

1 30 cc. burette, graduated to tenths, (burette holder if desired), with pinch-cock and delivery tube.

2 oz. alcoholic solution of turmeric.

2 qts. 95 per cent. alcohol to which ⅛ oz. dry carbonate of soda has been added and thoroughly shaken.

1 qt. caustic potash solution, of such strength that 31½. cc. exactly neutralize 5 cc. of a mixture of sulphuric acid and water, which contains 49 milligrams H_2SO_4 per cubic centimeter.

OPERATION

Take about two ounces of the clear alcohol and add a few drops of the turmeric solution, which should color the alcohol red, warm to about 150° Fahrenheit, then add 8.9 grams of the oil to be tested and shake thoroughly. The color of the solution changes to yellow. Fill the burette to the top of the graduation with caustic potash solution, and then run this solution from the burette into the bottle, a little at a time, with frequent shaking, until the color changes to red again. The red color must remain after the last thorough shaking. Now read off how many cubic centimeters and tenths of the caustic potash solution have been used, and this figure shows whether the material meets specifications or not.

To determine the free acid in tallow, everything is done exactly as above described, except that the tallow is melted before it is added to the alcohol.

Ten cubic centimeters of extra lard oil, at ordinary temperatures, and the same amount of melted tallow at 100° Fahrenheit, weigh almost exactly 8.9 grams. In ordinary work therefore it will probably not be necessary to weigh the oil or tallow. Measurement with a 10 cc. pipette, will usually be sufficiently accurate, provided the pipette is warmed to about 250° Fahrenheit, and allowed to drain, the last drops being blown out. In case of dispute, however, the balance must be used. (P. R. R. method).

Lard and tallow are very liable to have considerable amounts of free acid. The specification of purchase therefore generally states the limits of free acid permitted.

FREE ACID TEST.

(*Amer. Society for Testing Materials.*)

"About 10 g. of the oil are weighed into a 200 cc. Jena Erlenmeyer flask, 60 cc. of neutral alcohol added, the mixture warmed to about 60° C. and titrated with N/6 KOH, using phenolphthalein, the flask being frequently and thoroughly shaken.

"The result in the case of a mineral oil is usually reported in percentage of sulphuric anhydride (SO_3): with an organic oil, in percentage of oleic acid.

"It is suggested that it be reported, as with the saponification value, as the number of milligrams of KOH necessary to neutralize the acidity of one gram of oil."

Specifications for Lard Oil

Must be of the best quality and made from fresh lard; to be purchased and inspected by weight; the number of pounds per gallon to be determined by the specific gravity of the oil at 60° F. multiplied by 8.33 pounds, the weight of a gallon (231 cubic inches) of distilled water at the same temperature.

Oil will not be accepted which—

I. Contains admixture of any other oil.

II. Contains more acidity than the equivalent of 2 per cent. of oleic acid.

III. Shows a cold test above 42° F.

IV. Shows coloration when tested with nitrate of silver, as described below.

V. A half pint of the oil placed in an ordinary hand lamp without a chimney must burn with a clear, bright flame until 90 per cent. of the oil has been consumed; the lamp to be placed where it will not be affected by draft or air currents and the wick not to be touched during the trial.

1. *Test of Lard Oil.*—The cold test of oil is determined as follows: A couple of ounces of oil is put in a 4-ounce sample bottle and a ther-

mometer placed in it. The oil is then frozen, a freezing mixture of ice and salt being used if necessary. When the oil has become hard, the bottle is removed from the freezing mixture and the frozen oil allowed to soften, being stirred and thoroughly mixed at the same time by means of the thermometer until the mass will run from one end of the bottle to the other. The reading of the thermometer, when this is the case, is regarded as the cold test of the oil.

2. The nitrate of silver tests is as follows: Have ready a solution of nitrate of silver in alcohol and ether, made on the following formula:

Nitrate of silver	1 gram
Alcohol	200 grams
Ether	40 grams

After the ingredients are mixed and dissolved allow the solution to stand in the sun or in diffused light until it has become perfectly clear. It is then ready for use, and should be kept in a dim place and tightly corked.

Into a 50-cubic centimeter test tube put 10 cubic centimeters of the oil to be tested (which should have been previously filtered through washed filter paper) and 5 cubic centimeters of the above solution; shake thoroughly and heat in a vessel of boiling water fifteen minutes, with occasional shaking. Satisfactory oil shows no change of color under this test.

3. For the burning test an ordinary tin hand lamp to conform to the following description will be used:

Diameter of lamp at base, 3⅛ inches; height of cylindrical portion, 2¼ inches; height of top of burner from bottom of lamp, 2⅞ inches.

The burner will consist of two conical tubes placed side by side, each 1½ inches in length, fifteen sixty-fourths inch inside diameter at top, and eleven thirty-seconds inch inside diameter at bottom. The wick will consist of a sufficient number of threads of ordinary cotton lampwicking in each tube to make a properly fitting wick for lard oil.

Inspection and Delivery

1. Before acceptance the oil will be inspected. Samples of each lot will be taken at random, the samples well mixed together in a clean vessel, and the sample for test taken from this mixture. Should the mixture be found to contain any impurities or adulterations, the whole delivery of oil it represents will be rejected.

7. Maumene's Test

The rise of temperature produced when sulphuric acid is brought in contact with certain oils was first investigated by

Maumené, and the results of his experiments published in *Comptes Rendus,* **35**, 572.

When a mixture of oils has been analyzed and the components recognized, the proportions oftentimes can be determined by this reaction; that is to say, suppose the oil under examination to show a rise of temperature of 80° C., and the oils found by analysis to be lard oil and menhaden oil, their relative proportions can be determined by the following formula:

$$W_1 = W_3 \frac{t_3 - t_2}{t_1 - t_2}.$$

$$W_2 = W_3 \frac{t_3 - t_1}{t_2 - t_1}.$$

W_1 = proportion by weight of menhaden oil;
W_2 = proportion by weight of lard oil;
W_3 = weight of mixture;
t_1 = temperature of menhaden oil;
t_2 = temperature of lard oil;
t_3 = temperature of mixture.

The method is as follows:

Fifty grams of the oil are placed in a narrow tall beaker and 10 cc. of c.p. sulphuric acid added drop by drop with stirring and the rise of temperature during the operation noted.

Lard oil alone when treated with sulphuric acid gives a rise of temperature of 104° F.; menhaden oil, under similar conditions, a rise of 260° F. Using these values in the above formula we obtain 54.6 per cent. lard oil and 45.4 per cent. menhaden oil.

In the mixture containing a mineral oil mixed with animal, marine, or vegetable oil, the distinction would be even more pronounced, since the mineral oil shows but a very slight increase of temperature (generally from 35° F. to 41° F.). The increment of temperature would be dependent upon the other oil added to the mineral oil.

Briefly stated, the rise of temperature of the following oils would be:

	Name of Observer.				
	Maumené. °F.	Schaedler. °F.	Archbutt. °F.	Allen. °F.	Stillman. °F
Lard oil	104	....	....	105	102.2
Tallow oil	105–109	....	....	....	102
Neat's foot oil	113	122	109	....	104
Oleo oil	....	....	98	100	98
Elain oil	....	....	....	....	100
Sperm oil	....	....	123	113–116	118
Whale oil	....	....	197	195	197
Menhaden oil	....	....	253–262	258	262
Dog-fish oil	....	....	....	....	176
Cod liver oil	215–217	217	....	235	230
Crude cotton-seed oil	....	156	158	152–156	165
Rape oil	136	....	....	....	140
Castor oil	116	118	114	149	113
Olive oil	107	109	105–113	105–109	107
Rosin oil	....	82	....	64–71	50
Mineral lubricating oil	....	....	....	37–39	37
Earth nut	152	152	116–140	....	....
Sea elephant	....	....	....	....	149
Corn oil	....	....	....	....	185

Attention is drawn to the differences in the determination in rosin oil.

Rosin oil of the first run is a white, opaque, thick liquid containing all of the water of the rosin from which it is distilled, and it is this water that causes the rise of temperature above 10° when the oil is mixed with the sulphuric acid.

Rosin oils of the second and third runs are clear, limpid, dark red colored fluids, practically free from water, and when treated with sulphuric acid do not indicate more than 50° F. rise of temperature.

From these tests it is concluded that both Schaedler and Allen tested rosin oil that was a mixture of the first and second runs, or of an oil not properly separated into the different distillates.

Color Reactions of Oils with Nitric and Sulphuric Acids

Of the many color tests introduced for the identification of simple oils, preference is given to Heidenrich's sulphuric acid test and Massie's nitric acid test.

The color reactions of Chateau[1] in which barium polysulphide, zinc chloride, stannic chloride, phosphoric acid, and mercuric nitrate, in solutions, are used, while very interesting, seldom are of any advantage over the two tests noted above. Glassner's[2] nitric acid reactions are practically the same in results as Massie's, so that no advantage would be obtained in including the former.

Heidenreich's test is as follows:

A clear glass plate is placed over a piece of white paper, ten drops of the oil under examination are placed thereon, and one drop of concentrated sulphuric acid is added.

The color produced when the acid comes in contact with the oil is noticed as well as the color produced when the two are stirred with a glass rod. Many oils give off characteristic odors during the reaction, especially neat's foot oil, whale oil, and menhaden oil.

Massie's test is thus performed:

Nitric acid of specific gravity 1.40, free from nitrous acid is mixed in a test-tube with ⅓ its volume of the oil, and the whole agitated for two minutes.

The color of the oil after separation from the acid is the indication.

In mixtures of oils, the characteristic colors produced, by either Heidenreich's or Massie's test are often clouded, and in many instances no inferences can be drawn, yet with single oils the reactions are often distinctive and sufficiently strong to give confirmatory results.

In cod-liver oil, or whale oil, when mixed with a mineral or even vegetable oil, the characteristic brilliant violet color produced with sulphuric acid cannot be mistaken. This color, due to the presence of cholic acid, is found in most of the fish oils, but is much more pronounced in cod-liver oil.

The following table will indicate the colors produced by Heidenreich's and Massie's test:

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[1] Spon's Encyclopedia, 4, 1472-1475.

[2] *Chem. Centrbl.* 1873, 57.

	Heidenreich's test.		Massie's test.
	Before stirring.	After stirring.	
Lard oil	Yellow	Brown	Yellow
Tallow oil	Yellow	Orange	Colorless
Neat's foot oil	Yellowish	Red-brown	Red
Oleo oil	Colorless	Orange	Pink
Elain oil	Light green turning to brown	Brown	Orange-red
Sperm oil	Brown with purple streaks	Reddish brown	Red
Whale oil	Red-violet	Violet brown	Red
Menhaden oil	Red	Brown	Dark red
Dog-fish oil	Violet	Dark brown	Orange
Codliver oil	Red-violet	Dark brown	Orange-red
Crude cotton-seed	Brilliant red	Brown	Brown
Ref'd cotton-seed	Reddish brown	Red	Orange-red
Rape oil	Yellow-brown	Brown	Orange
Castor oil	Light yel. to brown	Pale brown	Orange
Olive oil	Light green	Greenish to light brown	Yellow to greenish
Rosin	Brown	Brown	Orange
Earth nut oil	Yellow to orange	Greenish	Reddish

8. Separation of Mineral Oil from a Vegetable or Animal Oil

Ten grams of the oil are weighed out in a dry weighed beaker (250 cc.), and to it are added 75 cc. of an alcoholic solution of potash (60 grams of potassium hydroxide to 1,000 cc. of 95 per cent. alcohol), and the contents evaporated until all the alcohol is driven off. In this process, if any animal or vegetable oil is present, it is formed into a soap by the potash, while the mineral oil is unacted upon. Water (75 cc.) is now added and the material well stirred to insure complete solution of the soap, and then it is transferred to a separatory funnel (Fig. 21), 75 cc. of sulphuric ether added, corked, the liquid violently agitated and allowed to stand for twelve hours. Two distinct liquids are now seen, the lower, the solution of the soap, the upper, the ether solution (colored, if mineral oil is present, colorless, if not). The aqueous solution is drawn off in a No. 3 beaker, the ethereal solution remaining in the separatory funnel. The former is placed on a water-bath, heated for half an hour, and until all traces of ether (which is absorbed by the water in a very small amount) is gone.

The solution is allowed to cool, diluted somewhat with water, and made acid with dilute sulphuric acid. Any animal or vegetable oil present will be indicated by a rise of the fatty acids to the surface of the liquid. (In this reaction the sulphuric acid decomposes the soap, uniting with the potash to form sulphate of potash and liberating the fatty acids of the oil.)

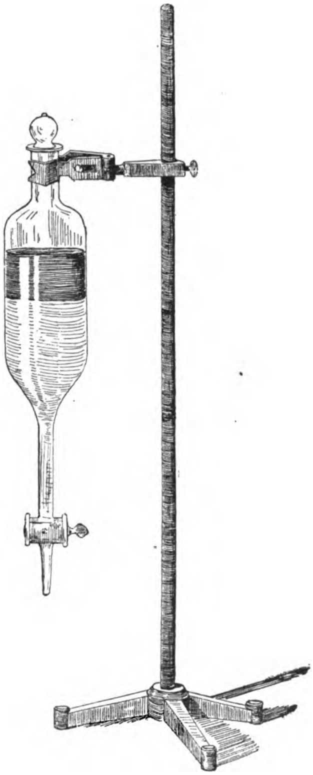

Fig. 21.

If it be desired to weigh the fatty acids, proceed as follows:

Weigh carefully about 5 grams of pure white beeswax, place it in the beaker upon the surface of the oil and water, and bring the contents nearly to boiling; the melted wax and fatty acids unite; allow to cool, remove the wax, wash with water, dry between folds of filter-paper, and weigh. The increase in weight of the wax over its original weight gives the weight of the fatty acids of the animal or vegetable oil in the lubricating oil.[1]

Another method of determining the weight of the fatty acids after saponification is given on page 59.

The weight obtained must be multiplied by the factor 0.97, since the fatty acids exist in the oil as anhydrides and not as hydrates, the latter being the form in which they are weighed.

Instead of weighing the animal or vegetable oil, some chemists prefer to make use of the ether solution, determining the hydrocarbon oil directly; in which case proceed as follows:

After drawing off the soap solution from the separatory funnel the ether solution is run into a weighed flask (about 250 cc.), and the ether distilled off. The residue in the flask now consists of the mineral oil and some water.

It is quite difficult to get rid of all this water. Direct heating is inadmissible, since the water spurts up through the oil out of the flask and is lost. This can be overcome by placing a glass tube through the stopper, in shape of the letter S. Any oil ejected against the tube or cork cannot escape, but returns to the base of the flask, while the heat is gradually increased in the flask and the water vaporized and passed out through the tube; three or four weighings are generally required before a constant weight is obtained. The former process is preferable, since it is performed much more rapidly than the latter, and also the animal or vegetable oil is positively shown, and generally can be identified; also many lubricating oils contain as high as 20 per cent. of hydrocarbon oil, volatile at or below 212° F. It is, of course, in the ether solution, and when the water is expelled from the oil, after the ether has been driven off, a large proportion of the volatile hydrocarbon is also evaporated. If now the animal or vegetable oil is not also determined, a serious mistake would be made; viz., reporting 20 per cent. of animal oil when it was volatile mineral oil.

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[1] Determination of Soap in Lubricating Oils, *J. Soc. Chem. Ind.*, 1896, p. 382.

The fatty acids in another sample of the oil are separated and subjected to qualitative test for identification of the oil from which they are derived. These tests comprise determination of melting-point, and congealing-point, page 10, color reaction with nitric and sulphuric acid, iodine absorption, and Maumené's test (rise of temperature upon addition of sulphuric acid).

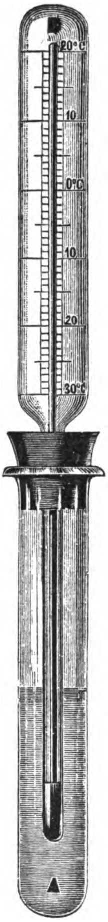

Fig. 22.

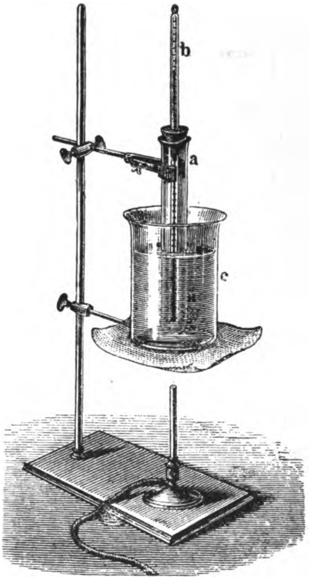

Fig. 23.

There are several methods of determining the melting-point of the fatty acids. Where a considerable amount of the fatty acids is available for experiment, the apparatus in Fig. 22 can be used. The glass cylinder is filled one-half with fatty acids, the cylinder closed with a rubber stopper, through which a thermometer is inserted, the bulb of which is covered by the fatty acids.

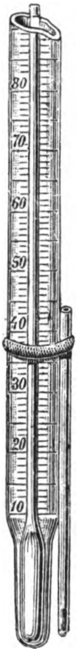

Fig. 24.

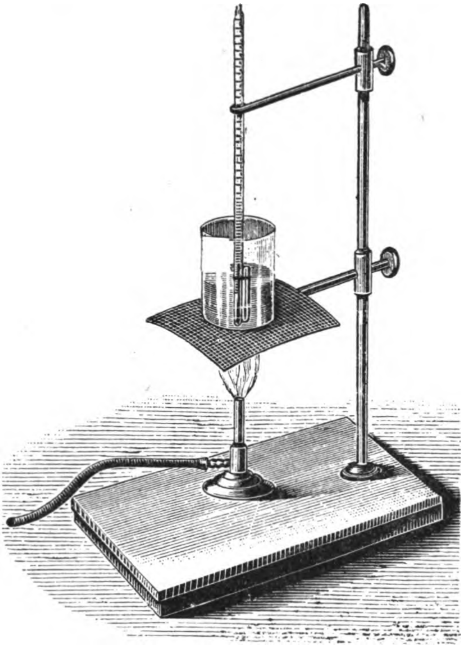

Fig. 25.

The apparatus is supported in a beaker containing water, as shown in Fig. 23.

If the fatty acids are liquid at ordinary temperatures, the water in the beaker must be cooled with ice until the fatty acids are congealed. The ice is removed, and the water gradually warmed until the fatty acids become melted. At this point the temperature is taken and recorded. Greater delicacy in the determination of the melting-point is obtained by using a small glass tube, sealed at one end. The liquid fatty acids are placed in this tube, then congealed, the tube then tied to a thermometer, Fig. 24, and both inserted in a beaker of water, as shown in Fig. 25. Another method is to cover the thermometer bulb with a layer of the solid fatty acids, about 3 mm. thick and immersing it in water; gradually heat the water and notice the temperature at which the fatty acids leave the thermometer bulb and ascend through the water.

TABLE OF MELTING-POINTS AND CONGEALING-POINTS OF FATTY ACIDS.

Fatty acids.	Melting-point. Degrees C.	Congealing-point. Degrees C.
Cotton-seed oil	33.0	30.5
Olive oil	26.0	21.0
Rape-seed oil	20.0	12.0
Castor oil	13.0	30.0
Sesame oil	26.0	32.0
Cocoanut oil	24.5	24.0
Lard	44.0	39.0
Tallow	45.0	42.0
Wool fat	42.0	40.0
Palm oil	48.0	43.0
Corn oil	20.0	14.0

The oils made use of in lubrication can be reported into two groups: saponifiable and unsaponifiable. To the former belong all the fatty oils; to the latter the mineral and rosin oils.

The method of Lux[1] is made use of to determine if any fatty oils are present in a mineral oil.

If rosin oil is suspected to have been added to the mineral, it can be identified by the Liebermann-Storch[2] reaction, or the process of E. Valenta[3] can be used.

The Liebermann-Storch reaction for detection of rosin oils:

One to two cc. of the oil under examination are shaken with acetic anhydride at a gentle heat; after cooling, the acetic anhydride is drawn off by means of a pipette, and tested by adding one drop of concentrated sulphuric acid. If rosin oil is present, a fine violet (fugitive) color is immediately produced. This test is thoroughly reliable for the detection of rosin oil in mineral oil.

These three tests will indicate, qualitatively, the presence of any fatty or rosin oil in a mineral oil. It is rarely, in the better class of lubricating oils, that more than one oil is added to a mineral oil, such, for instance, as lard oil, or tallow, in which case saponification easily separates the two oils, and identification of each by special tests can be made.

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[1] *Ztschr. anal. Chem.*, **24**, 347.

[2] Benedikt and Lewkowitsch's "Oils, Fats, and Waxes," p. 179.

[3] *Ztschr. anal. Chem.*, **25**, 441.

Twenty grams of the oil are weighed in a No. 3 beaker, 100 cc. of an alcoholic solution of potash (80 grams potassium hydroxide to 1 liter alcohol of 98 per cent.) are added, and heat applied with stirring until the alcohol is all driven off; add 100 cc. water, heat with agitation, *cool*, add 50 cc. ether, transfer to separatory funnel, stopper, shake well and allow to stand two hours. Draw off the soap solution.

1. **Soap Solution** (Containing the fatty acids of the lard and cotton-seed oils). Heat ten minutes nearly to boiling, cool, acidify with dilute sulphuric acid, allow to stand a few hours; collect the separated fatty acids; determine their weight, then test as follows: First portion: Determine the "melting point." Second portion: Determine the iodine "absorption" and their rates by formula: $x = (I - n)$ $x = m - n$	2. **Ether Solution** remaining in the separatory funnel is transferred to a flask, the ether distilled and the mineral oils weighed.

When, however, the oil added to the mineral oil itself contains an adulterant, such as lard oil to which cotton-seed oil has been added, then the fatty acids separated by saponification will require a more extended examination to prove the presence of both lard oil and cotton-seed oil.

The preceding skeleton scheme is given to show the application of the above upon a lubricating oil that qualitative analysis has shown to contain mineral oil, lard oil and cotton-seed oil.

There are several methods for the quantitative determination of the amounts of vegetable and animal oils when mixed with each other or when the mixture is incorporated with a mineral oil. The determination of the iodine absorption is the most delicate and correct provided no fish blubber or olive oils are present.

If the fatty acids have been separated by saponification, from a mineral oil, this iodine value can also be determined.

The method of Salkowski[1] depends upon the fact that vegetable oils (except olive) contain phytosterol and that animal fats (butter excepted) are free from it, containing cholestrol, the latter not being present in vegetable oils.

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[1] Benedikt's "Oils, Fats, and Waxes," p. 255.

Fifty grams of the sample are saponified with alcoholic potash; the soap solution is diluted with a liter of water and 250 cc. of ether added. When the two layers have separated, the aqueous layer is run off and the ethereal liquid filtered, and evaporated to a small bulk. To insure complete absence of unsaponified fat, it is best to saponify again with alcoholic potash and to repeat the exhaustion with ether. The ethereal layer is then washed with water and the ether evaporated in a deep basin. The residue is next dissolved in hot alcohol, the solution boiled down to 1 or 2 cc. and the residue allowed to cool. If phytosterol or cholesterol be present, crystals will separate out. They are dried on unglazed porcelain and their melting-points determined.

The saponification value of oils is often made use of for identification; but as this value varies with the age of the oil, it is extremely difficult to obtain concordant results, and as the majority of oils have a saponification value of 193, excepting rape-seed oil and castor-oil, which are lower, it cannot be relied upon. It, however, is of value in determining the amount of liquid waxes in the presence of oils.

GUMMING TEST.

This test gives an indication of the amount of certain changes that may be expected in a mineral oil when in use. These resinified products, resulting from use, increase the friction of the revolving or rubbing surfaces.

It is also a measure of the amount that an oil will carbonize in a gas or gasoline engine cylinder. It is applied after the manner of the elaidin test, by thoroughly mixing together in a cordial glass 5 grams of the oil with 11 cc. of nitrosulphuric acid and keeping the mixture cooled in a pan of water at 10° to 15° C. Brownish spots or, in case of bad oil, masses, form around the edges and become red in the course of two hours.

As has been shown by long experience, the oil showing the least tar or gum is the best oil; it also absorbs the least oxygen.

The nitrosulphuric acid is made by saturating sulphuric acid of 1.47 specific gravity cooled to 0° C., with nitric oxide (NO)." (*Amer. Soc. Testing Materials.*)

SULPHUR TEST.[1]

Proceed as follows: A portion of a sample, 0.7 to 1.0 g., is burned in a calorimetric bomb containing 10 cc. of water and oxygen under a pressure of 30 atmospheres. A lower pressure sometimes gives inaccurate results. If the sample contains more than 3 per cent. of sulphur the bomb is allowed to stand in its water bath for 15 minutes after ignition of the charge. In case the sulphur content is as high as 5 per cent., oxygen under pressure of 40 atmospheres is used. With these high pressures in a Berthelot bomb of 500 to 600-cc. capacity, repeated trials have failed to show even traces of carbon monoxide or sulphur dioxide. If a smaller bomb of about 175-cc. capacity, such as the Peters or Kröker, is used, incomplete combustion from a lack of oxygen may result if too large a sample is taken.

After cooling,—15 minutes is usually enough,—the bomb is opened and its contents are washed into a beaker. If the bomb has a lead washer, 5 cc. of a saturated solution of sodium carbonate is added, the contents are heated to the boiling point, boiled for 10 minutes, and are then filtered. This operation is necessary to decompose any lead sulphate from the washer. The united washings are then filtered, acidified with hydrochloric acid, boiled to expel all carbonic acid, and the sulphuric-acid content is determined in the usual way with barium chloride.

Gravimetric determination is preferred to volumetric, because the nitrogen contained in the air originally in the bomb is oxidized in part to nitroacids, which cause a small error if the volumetric determination alone is used. The sulphur content of any combustible material, from light gasolenes weighed in a tared gelatin capsule to solid bitumens and cokes, can be readily determined by this method.

This method of burning in a bomb is accurate, practicable, and rapid, and is recommended in preference to all of the other methods there described. The calorimetric determination, if desired, can be made at the same time.

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[1] Allen and Robertson, Technical Paper No. 36, Bureau of Mines, p. 10.

TEST FOR WATER.[1]

Dilute the oil with an equal volume of benzene and whirl it vigorously in a centrifuge until the separated layer of water does not appear to increase in volume. However, as water is somewhat soluble in any diluent used and also in oils, a portion of the water content will fail to appear; consequently the method in which a diluent is used can not be considered accurate. It is advisable first to agitate the diluent vigorously with water and then to separate with the centrifuge in order to saturate it with water before using.

Groschuff[2] states that 100 g. of benzene will dissolve 0.03 g. of water at 3° C. and 0.337 g. of water at 77° C., whereas petroleum products (density 0.792) will dissolve from 0.0012 g. at 2° C. to 0.097 g. at 94° C.

Alternate Method.—The water content may be accurately and conveniently determined during the course of an ordinary distillation in the following manner:

Two hundred grams of the sample are weighed into a one-fourth-liter distilling flask and the distillation carried out in the ordinary manner at the rate of one drop of distillate per second. The distillation can be performed most accurately in an electric still. At temperatures between 90° and 150° C. the water distills over and can be removed from the receivers by means of a micropipette and weighed. Usually a few drops of water adhere to the condenser and fail to run into the receivers; in this event a small pellet of absorbent cotton, moistened with water, squeezed as dry as possible, and weighed, is fastened to a wire and run up into the condenser to remove these last traces of water. The increase in weight of the cotton pellet, figured at water, is added to the weight of the water in the receivers.

With an oil containing considerable water, it is advisable to cause a slow current of dry, inert gas, such as carbonic acid, to bubble through the oil in the distilling flask to carry off the vapors of oil and water as soon as formed. The gas current will reduce bumping and the overheating of the oil to a minimum. The condenser must also be kept well cooled throughout the distillation. This method is accurate to less than 0.03 per cent.

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[1] Allen and Jacobs, Technical Paper No. 25, Bureau of Mines, p. 5.

[2] E. Groschuff, "The Solubility of Water in Benzene, Petroleum, and Paraffin Oil," *Chemical Abstracts*, Vol. 5, p. 2550 (Aug. 10, 1911).

GASOLINE TEST.

Dissolve 10 cc. of the oil in 90 cc. of 86° to 88° gasoline (from Pennsylvania crude) in the graduated tube[1] shown in Fig. 26.

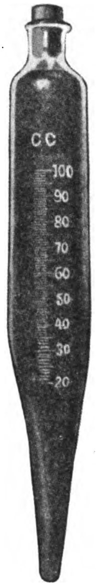

Fig. 26.

Allow to stand one hour at 70° F.; not more than 5 per cent. of flocculent or tarry matter should have settled out. If the test is first applied to the oil before making the flash test and again after this test, it shows the extent to which the oil is changed upon heating. Other things being equal, the oil which is changed the least is the best oil.

MICROSCOPICAL EXAMINATION.

Put a few drops of the well-mixed oil on a slide and note the nature of the suspended matter—whether carbonaceous specks, flakes of paraffin, which disappear on warming, or foreign matter. Polarized light is a great aid in detecting paraffin crystals, showing them white on a black background. The polariscope is excellent for this same purpose, showing them when it is impossible to see them with direct light.

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[1] The flat tube originally proposed by Conradson cannot be obtained in the market.

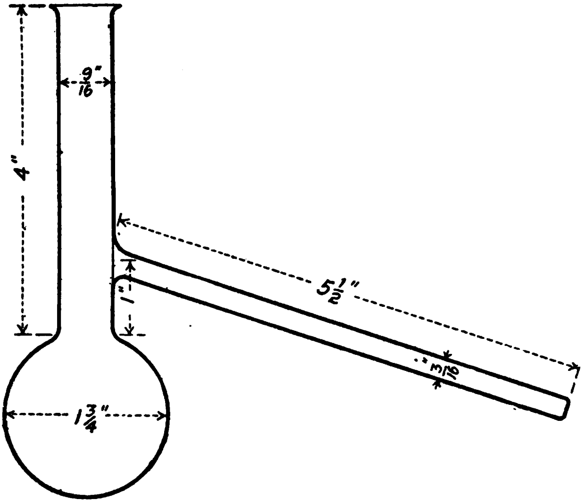

Fig. 27.—"Gray" carbon residue flask.

CARBON RESIDUE TEST.

Gray's Method.—To a tared 1-oz. flask of the dimensions shown in Fig. 27, add 25 cc. of the oil to be tested and weigh. Wrap the neck of the flask with asbestos paper as far down as the side arm. Stopper tightly with a good cork. Connect to a small aerial condenser by plugging the space with asbestos or glass wool. Provide a shield which will protect the flame and the flask up to the side tube. Using the flame of a good Bunsen burner, heat the flask so that the first drop of distillate will come over in approximately five minutes. Continue the distillation at such a rate that one drop per second will fall from the end of the condenser. As the end of the distillation approaches, increase the heat just enough so that no heavy vapors are allowed to condense and drop back into the flask, continue increasing the heat until the flask is enveloped in the flame, and hold the temperature five minutes. Allow the flask to cool, remove the asbestos covering and cork, and burn out completely the carbon and oil in the neck as far down as the side tube, and in the side tube. Heat the bottom of the flask until no more vapors are given off. Cool and weigh.

FIXED CARBON IN OIL.

Residues—Petroleum, Pitch, etc.—This is done in the same manner as the determination of Fixed Carbon in coal, as described in *Engineering Chemistry* (Stillman), 4th Edition, page 2.

ESTIMATION OF PARAFFIN IN MINERAL OILS.

The following method is due to Holde (after Engler and Bohm):

Ten to 20 cc. of oils poor in paraffin (Russian distillates, etc., setting below —5° C.), or 5 g. of such as are rich in that constituent (American, Scotch, or Galician oils setting at or above 0° C.), are treated, at the ordinary temperature, with a mixture of 98.5 per cent. alcohol and anhydrous ether (1:1) until a clear solution is obtained. The liquid is cooled in a freezing mixture of ice and salt to about —20° C., when more alcohol-ether is gradually added, with thorough agitation, until no oil drops, but only solid paraffin flakes or crystals remain in suspension, and then, while still cooled to at least —19° to —21° C., the liquid is poured on to a chilled 9 cm. filter paper, previously moistened with alcohol-ether mixture, which is contained in the apparatus shown in Fig. 72. The precipitate is washed with cold (—19° to —21° C.) alcohol-ether (1:1); or for soft paraffin (2:1) at a temperature as much below —15° C. as possible. In the case of soft paraffin, the temperature should average —18° to —19° at the highest. In washing the precipitate it is repeatedly stirred up, and as soon as 5-10 cc. of the filtrate leaves on evaporation only a trace of fatty or paraffin-like residue, solid and not oily at the ordinary temperature, the washing is discontinued.

If any doubt exists as to the paraffin being thoroughly freed from oil, or if the washing takes too long, the filter should be removed to another funnel, and the contents dissolved into a small flask with the least possible quantity of benzine. After evaporation of the benzine, the paraffin is redissolved in 4 to 5 cc. of warm ether, which is then mixed with twice its volume of absolute alcohol, vigorously stirred, and cooled to —18° to —20° to reprecipitate the paraffin, which is again filtered and washed, as already described, until free from oil. This reprecipitation is necessary for oils containing much soft paraffin, otherwise so much liquid is used in washing the precipitate that an appreciable quantity of paraffin is dissolved. The purified paraffin is finally dissolved into a tared flask with hot benzine or ether, which is distilled off, and the residue is heated on the steam-bath until the smell of benzine or ether has disappeared. The flask is then heated inside the water-oven for ¼ hour and weighed when cold. Prolonged heating causes loss of paraffin. The whole operation occupies from one to two hours. Duplicate results with the same sample agree within 0.23 per cent. for hard paraffin, and 0.33 per cent. for soft paraffin. Two samples of Russian machine oil yielded 0.34 per cent. and 0.36 per cent. of paraffin respectively. An American spindle oil, fluid but thick at 2° C., and which set at 0° C., was found to contain 4.11 per cent. of paraffin.

SOAP TEST.

The test depends upon the fact that the metaphosphates of the earthy and alkali metals and aluminum are insoluble in absolute alcohol. Five to ten cubic centimeters of the oil are dissolved in 5 cc. of 86° gasolene or ether, and 15 drops of a saturated solution of "stick phosphoric acid" in absolute alcohol are added, shaken and allowed to stand: the formation of a flocculent precipitate indicates the presence of soap. For the accurate determination of these soaps a known quantity of the oil must be ignited and the residue quantitatively examined.

Saponification Value.

This is expressed by the number of milligrams of potassium hydrate necessary to saponify one gram of the oil. From 2.5 to 10 grams of the oil, according as 65 to 20 per cent. of saponifiable matter are supposed to be present, are boiled with 25 cc. N/2 alcoholic potash in a 200-cc. Jena Erlenmeyer flask. A reflux condenser is used and the boiling may require from 5 to 8 hours. The excess of alkali is titrated with N/2 HCl, using phenolphthalein. The strength of the N/2 KOH is determined by boiling 25 cc. in similar flasks alongside of those in which the oil is treated and for the same length of time.

Alcohol purified with silver oxide according to Dunlap's method[1] should be used as well as KOH, purified by alcohol. For heavy oils, dissolve them in 50 cc. of C. P. benzol before adding potash.

Determination of Tarry Matters in Petroleum Products.

At present the French volumetric method is exclusively used, and is very simple in its principle and practical application. According to this method the quantity of tarry matter in any petroleum product is judged by the increase in the volume of the sulphuric acid or the decrease in the volume of the tested product, which is, after being diluted with benzine, submitted to the action of sulphuric acid, which carbonizes and dissolves the tarry substances. This method is, as already mentioned, very simple and easy in practice, but is connected at the same time with a serious source of errors. Besides the principal action of carbonizing and extracting the tarry substances, sulphuric acid also extracts unsaturated hydrocarbons, polymerises others, etc. Thus, for instance, a simple experience will show that even perfectly well refined oil treated with sulphuric acid decreases in volume up to 8 per cent. But the incorrectness due to this may be obviated. Thus the quantity of unsaturated hydrocarbons extracted by the sulphuric acid can be ascertained by determining the iodine value, and this can then be calculated to an equivalent value in sulphuric acid, according to the very simple formula, $V = \frac{49T}{127d}$, where V is the volume of sulphuric acid, T the iodine value, and *d* the specific gravity of the sulphuric acid. In the works, where a great number of tests are continually to be made, the operation becomes very simple, as *d* —the specific gravity of the sulphuric acid in use—is practically a constant, and so is the iodine value T for a whole series of products.

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[1] *Journal Amer. Chem. Soc.*, Vol. 28, p. 397.

Greases

Horace W. Gillett in the *Journal of Industrial and Engineering Chemistry*, June 1909, states as follows:—"Commercial greases may be divided into the following classes:

"A. The tallow type: these greases are made of tallow and more or less of an alkali soap, commonly the sodium or potassium soaps of palm oil, mixed with a smaller amount of mineral oil. These were the principal types of lubricating grease ten or twenty years ago, but to-day are less used than the greases of type B.

"B. The soap-thickened mineral oil type: these are the most common journal greases of to-day, and are composed of mineral oil of various grades made solid by the addition of calcium or sodium soaps. Calcium soap is more used than sodium soap.

"C. Types of A or B with the addition of a mineral lubricant —usually graphite, mica or talc.

"D. The rosin-oil type: these consist of rosin oil thickened by lime, or less commonly, litharge, to which is added more or less mineral oil, either paraffin or asphalt oils being used.

These greases are sticky, usually contain 20 to 30 per cent. of water, and find their chief application as gear greases where true lubrication is not so essential as prevention of wearing and rattling of the gears.

Some very heavy bearings are occasionally lubricated with this type of grease. Tar, pitch, graphite and such fillers as wood pulp and ground cork are often put into these gear greases.

"E. Non-fluid oils: these are thin greases stiffened to some extent with aluminum oleate or a mixture of soaps, as sodium and calcium.

COMPOSITION OF SOME GREASES.*

Name.	Flash pt. °C.	Mlt. pt. °C.	Consistance. Grams 20° C.	Water.	Ca. soap.	Filler.	Min. oil.	Fatty oil.	Free acid.	Coeff. friction.
Graphite	195	93	18	tr.	11	16	56	17	0	0.097
Summer motor	160	87	170	tr.	38	..	36.5	25	tr.	0.075
Winter motor	175	86	7	tr.	23	..	40	37[5]	6.1	0.063
K_1	193	85	24	0.2	16	..	67	16	0	0.057
K_2	195	93	66	0.3	20	..	60	20	0.3	0.054
Auto	190	79	11	1.0	19	..	60	20	tr.	0.046
Tallow	210	52	150†	2.5	..	1.4[1] 2.1[1]	22	73.5	0	0.022
Tallow xx	215	49	200	tr.	..	30[7]	20	48	0	0.029
Lead resin oil	240	102	7	24.7	..	1.7[2]	..	0	0	0.067
Lime resin oil	198	77	31	tr.	..	9.9[3]	..	0	0	0.048
Lime resin oil	198	75	4	20.0	..	7.8[3]	..	0	0	0.036
Soda grease	215	83	35	0	..	22[4]	78[6]	0	0	0.019
Non-fluid oil	210	76	27	0	9.8	12.9[4]	70.3	7	0	0.026
No. 4 petrolatum	247	47	6	0	..	..	100	—0	0	0.018
Lard oil	265	5	0	0	..	..	0	100	..	0.011

* Gillett

† Estimated.

[1] Potash soap.

[2] Lead soap.

[3] CaO.

[4] Soda soap.

[5] Mainly palm oil.

[6] Oil of 24.2° Be.

[7] Paraffin,

"F. Special greases, such as a mixture of wood pulp and graphite, thin greases of any of the above types mixed with wool or cotton fibers, hot neck greases, freak greases containing rubber, etc."

The analysis of a lubricating grease may have one or two objects in view:—to duplicate the grease, or to determine its value as a lubricant. Without resorting to mechanical tests of the actual friction reducing power of the grease in question, the first is probably the easier problem. In the analysis of a grease, we may cover the following points:

Consistency.
Melting point.
Flash point.
Content of free acid.
Amount and nature of soap.
Ash, not alkali, from soap, nature and amount.

Wool grease[1] is used to some extent in the cheaper grades of lubricants, the consumption for this purpose increasing yearly. It is partially unsaponifiable and, if present, will be found in the ether extracts with the mineral oil, and also in the soap solution, in the analysis as usually conducted of a mixed lubricating oil.

R. Krause, of Wiltemberger, proposes a lubricant using wool fat, as follows: An emulsion of wool fat in soda solution is precipitated with concentrated alum solution, whereby is obtained a brown, soft, tenacious mass of aluminum lanoleate. This is freed from salts and soap by pressing and washing with hot water, then dried and finally added to mineral oils which it is desired to thicken for use as lubricants, solution being effected by heat. One part of aluminum lanoleate mixed with fourteen parts of light Scotch mineral oil with a gravity of 0.855 to 0.860 yields a lubricating oil with the viscosity of olive oil.

Degras or sod oil is a waste product obtained in the chamoising process. It is largely derived from whale oil or poor quality of cod-liver oil used in chamoising.

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[1] For method of analysis of wool grease ("Yorkshire grease") and lanoline, consult Benedikt and Lewkowitsch, pp. 582-585.

Gill, "Oil Analysis," pp. 141-147.

The English-German method of treating skins produces sod oil as a waste product. The French method produces degras. These fats are largely used in the production of cheaper lubricants.

Bone fat is made use of in lubrication mixed with mineral oils. It is recovered from bones either by boiling with water or extracting with solvents. It does not readily become rancid. Its examination is made similarly to that of tallow.

Coefficient of Friction

The ratio of the force required to slide a body along a horizontal plane surface to the weight of the body is called the coefficient of friction. It is equivalent to the tangent of the *angle of repose,* which is the angle of inclination to the horizontal of an inclined plane on which the body will just overcome its tendency to slide. The angle is usually denoted by ϕ, and the coefficient by f. $f = \tan \phi$. (Kent.)

Of the various machines used for this purpose nearly all are deficient in conducting tests under extreme pressure. However, as all the tests are relative, an idea of the value of a lubricant can be formed by a series of comparative tests upon the same instrument.

G. B. Heckel thus describes the Thurston and Henderson-Westhoven machine: The primary idea of determining durability is to determine how much rubbing a lubricant will withstand before exhaustion of its power to maintain the friction at some agreed minimum. For this there is no device superior to the Thurston oil-tester, in which a pair of brasses are forced against a journal in opposite directions by a spring being lodged in a pendulum which is free to swing about the journal, the friction being measured by the inclination to the vertical of a line joining the center of the journal and the center of gravity of the pendulum. The defects of this machine lie in the infinitely variable rate of metallic wear between rubbing surfaces, which contaminates the oil before it has been exhausted, as well as in the escape of the lubricant between the surfaces.

These imperfections have been overcome in the Henderson machine or the so-called Henderson-Westhoven machine, a modified Thurston tester. (Figs. 28 and 29.)

With this machine lubricants can be tested at the same moment for the degree of heat developed in the bearing surfaces as well as their friction-reducing qualities.

The journal, A, rests upon the supporting beds, BB, and is revolved by the pulley, C. This journal, A, extends on both sides beyond the supports, BB, and the projecting ends are embraced by brass boxes, DD, to which are fastened the pendulum parts, EE. Strong spiral springs, *mm,* in the interior of the pendulum arms, force the lower pair of brasses, DD, against the journal, A, and the pressure of these springs may be regulated by means of the screw, N. A pointer attached to the movable block, *o,* indicates on the scale, P, as in a spring balance, the thrust of the spring against its bed, in kilograms per cubic centimeter. By the revolution of the journal, A, the swinging arms, EE, are actuated by friction in the direction of the motion, and the degree of their deviation from the vertical is read by means of the pointers, FF, on the quadrants, GG. On many machines the scales give, besides the deviation, also the coefficient of friction which has been calculated from the former.

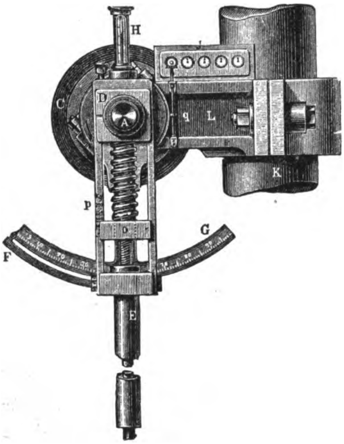

Fig. 28.

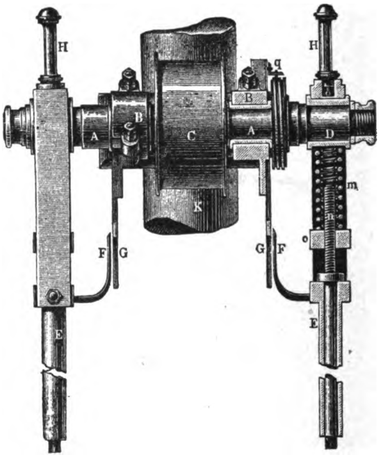

Fig. 29.

In the upper brasses, DD, a thermometer, H, is fixed to show the degree of heat developed by the friction, and the revolution counter, J, actuated through the endless screw, *q*, records the revolutions of the journal, A. The column, K, through its two arms, L, carrying the boxes, BB, serves to support the entire device.

In operation the oil to be tested is introduced by means of a small glass tube or pipette, through an orifice in the upper brasses, DD, the journal having been thoroughly cleaned. The position of the thermometer and of the revolution counter are noted, and the journal is then put into motion with 200 or 300 revolutions per minute. At each succeeding five-hundredth or thousandth revolution the temperature and the degree of deviation of the pendulum arms, as shown by the quadrant, are noted, and when the friction has raised the temperature in the boxes

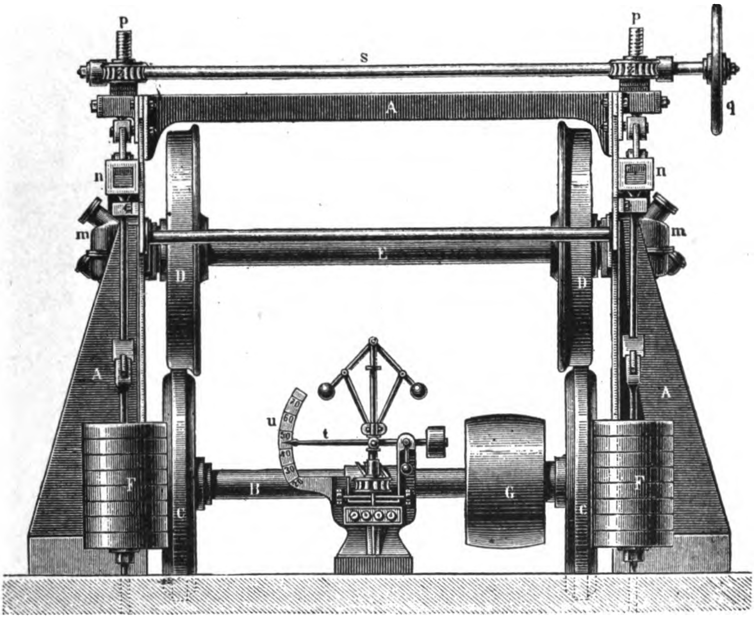

Fig. 30.

about 30° (usually in about half an hour) the machine is stopped. In figuring up results, the sample of oil which, with an equal rise in temperature at the point of friction, gives the slightest deviation of the swinging pendulum arm, and the greatest number of revolutions is regarded as the best. The advantages noted in this device are its facilities for testing materials under any pressure, even up to the load limit on a freight car axle; the number of data obtainable at one time; and the ease with which two simultaneous tests of competing oils can be made on the one machine.

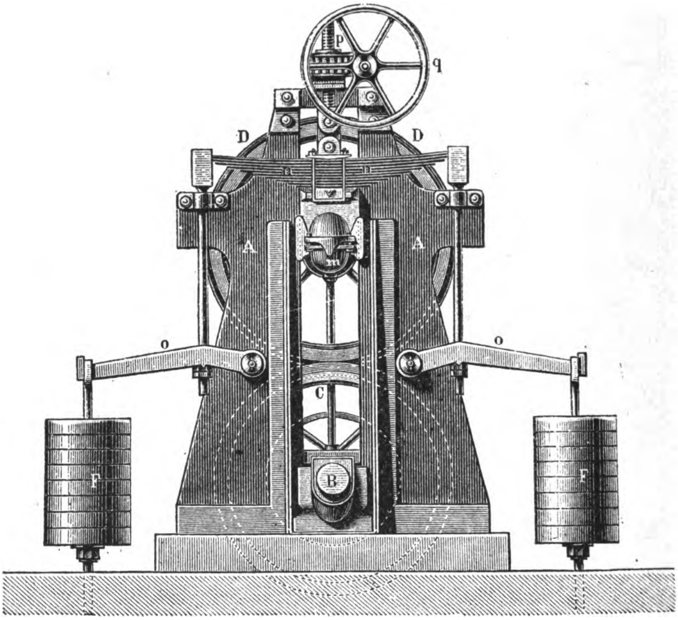

Fig. 31.

The apparatus used for testing lubricants by the officials of the Paris-Lyon Railway is shown in Figs. 30 and 31. Here the conditions are maintained as nearly as possible as would occur in railroad practice, the friction being determined by means of two freight-car wheels.

The heavy cast-iron frame, A, stands upon a firm stone foundation and carries the shaft, B, on which are fastened the two friction wheels, CC. These are placed at the same gauge as the railroad track. Two ordinary car wheels, DD, with axle, E, are placed above and in contact as shown in the figure. The car axle E, is fitted at each end and into the axle boxes, *mm*. The boxes have the same arrangement as those in the railroad cars and serve for the reception of the lubricant. Resting on each side of the axle boxes are the strong springs, *nn* (Fig. 30), on the end of which the weights, FF, work by means of the levers, *oo*. By taking off or putting on of weights, FF, E can carry any load desirable.

On the lower shaft is the driving wheel, G, also a screw by which the movement of the shaft is carried to a figured dial. This dial sets not only the index showing the number of revolutions but also the index needle, *t*, in motion which indicates on the scale, u, the approximate rapidity of the wheel-rims in kilometers per hour. The two friction wheels, *cc*, are turned eccentrically about 2.5 mm. that by the motion a weak vertical oscillation arises which is communicated to the upper wheels whereby the rattling of the wheels upon the car track is imitated.

In making a trial, the lubricant to be tested is placed in the thoroughly cleaned axle boxes, *mm*, the springs are lifted to the utmost release of the upper shaft, and the lower shaft is placed in rotation. Not until the whole is in motion are the springs brought down, and later loaded with the intended weight. The oil which by this test carries the burden with the greatest rapidity without heating of the axle-boxes is to be considered the best. By this apparatus it is possible to judge of the practical working of an oil or compounded oil, and especially if the car axles would become heated, a point of vital importance as regards the use of the lubricant.

Another instrument of a similar design is the Riehlé, in use in many railroad laboratories in the United States, for testing lubricants. The capacity is 20,000 pounds; it determines the coefficient of friction, the pressure per square inch of journal and records the temperature.

It consists of a Master Car Builder's axle journal, which is removable from the main spindle. This journal is made to revolve by cone pulley at different speeds, and in either direction, and can

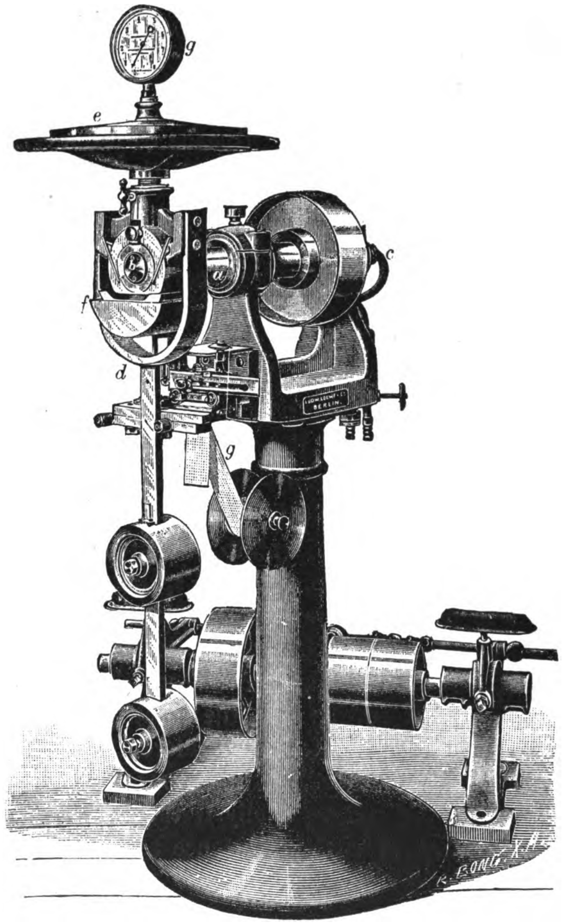

Fig. 32.

be loaded to different pressures per inch by means of the lever system. The oil can be supplied through a hole in the top, which is tapped to receive a sight-feed oiler, or funnel, or other arrangement.

The friction is weighed on the beams, which are arranged in double system to balance each other, allowing the machine to be run in either direction. The opening in the frame over the journal is made large enough to take a regular car box if desired.

The frame and beams can be raised by rope sling and hoist for change of journal, cleaning up, etc.

There is an end motion of about ¼ to ⅜ inch given to the axle by the gearing shown at the end, giving a natural movement like cars. The weighed end of spindle runs loose on large rollers, to avoid friction and heating.

The instrument in general use, for the friction tests of lubricants, in Germany, is the "Mertens-Machine," (Fig. 32).

In principle it is a modification of the Thurston.

It is fully described by D. Holde in his work—"Untersuchung der Mineralöle und Fette," Berlin, 1909.

An oil tested upon the tester may show a fine lubricant, while put under practical working upon a freight car (for instance) would prove vastly inferior. This very often happens, and it has led many engineers to test each oil by a long run, with the particular kind of machinery upon which it is to be used.

A record blank used by the engineers of the Baltimore and Ohio Railroad, for testing oils upon their locomotives, is given herewith. It is a point in instance. After experimenting months upon an oil its work is established so that a practical comparison can be made with other brands of similar composition for the same purpose.

Baltimore and Ohio Railroad

Subject..................... 191

....

Engineer of Tests.

DEAR SIR: Below please find report from locomotives inspected this day.

Engine No.	Train No.	Service	Engineer	Fireman	Left	Time	Arrived	Time	Miles run	Average speed, miles per hour	Condition of bearings	Kind of oil used		Miles run per pint of oil allowed		Miles run per pint of oil used		Number of drops per minute	
												Cylinder	Journal	Cylinder	Journal	Cylinder	Journal	Cylinder	Journal

..................................*Inspector.*

Chicago, Burlington and Quincy Railroad Company

CHEMICAL LABORATORY

AURORA, ILL.,..................191

To.......................... Supt. M. P.:

DEAR SIR: I have made an examination of sample of above oil, and have obtained the following results:

Flashing-point .... °F. Ash ....%. Tar ....%

Burning-point .... °F. Cold test ....at..........°F.

Specific gravity .... °B. Viscosity at.........°F. 100 cc. oil

Loss at.......°F. for 3 hours....% Flows from instrument in...seconds

FRICTION TEST ON THE THURSTON OIL TESTER

Date.	First trial.		Second trial.		Third trial.		Average.
Amount used.	Temp.	Arc.	Temp.	Arc.	Temp.	Arc.	
Highest reading	. . . .	. . . .	. . . .	. . . .	. . . .	. . . .	. . . . . .
Lowest reading	. . . .	. . . .	. . . .	. . . .	. . . .	. . . .	. . . . . .
Range of reading	. . . .	. . . .	. . . .	. . . .	. . . .	. . . .	. . . . . .
Average reading	. . . .	. . . .	. . . .	. . . .	. . . .	. . . .	. . . . . .
Time run in minutes	. . . .		. . . .		. . . .		. . . . . .
Total revolutions	. . . .		. . . .		. . . .		. . . . . .
Revolutions per minute . . .	. . . .		. . . .		. . . .		. . . . . .
Speed, miles per hour . . .	. . . .		. . . .		. . . .		. . . . . .
Pressure, total lbs	. . . .		. . . .		. . . .		. . . . . .
Pressure, lbs. per square inch	. . . .		. . . .		. . . .		. . . . . .
Coefficient of friction	. . . .		. . . .		. . . .		. . . . . .
Lubricating value, with Extra Lard Oil as 100	. . . .		. . . .		. . . .		. . . . . .

Received ....19..
Tested ....19..
Sample No. or Letter ....
Blank No. ....
Letter Book No. ......Page.......

Car No. and Initials................
Tank or No. of Barrels ....
Name of firm supplying ....
Price .... cents per gallon.

Yours truly,
......................Chemist.

For the............................R. R. Co.

Chicago, Burlington and Quincy Railroad Company

Specifications for Black Engine Oils

("Petroleum lubricating oils;" "well oils;" "petroleum stock oils;" or "passenger and freight car lubricating oils.")

Uses.—For lubricating the journals of passenger and freight cars and locomotives, and for miscellaneous lubrication.

Grades.—"Summer," "25 degree," "15 degree," and "zero."

Requirements.—For all grades:

Specific gravity, between 26° and 29° B.
Loss at 100° F. for three hours, not over ¼ per cent.
Flashing-point, for all but "zero" oil, not under 300° F.
Flashing-point, for "zero" oil, not under 250° F.
Burning-point, for all but "zero" oil, not under 375° F.
Burning-point, for "zero" oil, not under 300° F.
Cold Test—Summer oil must flow at 60° F. or above.
Cold Test—25° oil must flow at 30° F. or above.
Cold Test—15° oil must flow at 20° F. or above.
Cold Test—Zero oil must flow at 5° F. or above.

All these oils must be pure petroleum oils, free from other compounds, and from dirt, grit, lumps and specks; transparent and greenish or reddish (not black) in *tint,* when spread as a *thin* film on glass and looked through toward the light; translucent and greenish when held in a horizontal position. Preference will be given to those oils which are low in tarry matters and in ash, and which do not "froth" when tested for flash and fire.

Oils differing notably from above requirements will be rejected.

Chicago, Burlington and Quincy Railroad Company

Specifications for Cylinder Stock

Use.—For making cylinder lubricant. One grade.

Requirements.—Must have a flashing-point not lower than 475° F., a burning-point not lower than 575° F., and a specific gravity between 25°

and 28° B. Must not undergo a loss greater than one-half (½) per cent., when exposed for three (3) hours to a temperature of 350° F. Must be free from dirt, grit, lumps, and specks; transparent and greenish or reddish (not black) in tint, when spread as a thin film on glass and looked at toward the light.

Philadelphia and Reading Railroad Company

Specifications for Compound Oils

The specifications for compound oils, dated April, 1892, are hereby annulled and superseded by the following:

When a shipment of oil is received, a sample shall be taken at random and forwarded to the Test Department. This sample will be examined and the entire shipment accepted or rejected on its merits. If rejected, the shipment will be returned at the shipper's expense.

Cylinder Oil

This oil shall consist of a high-grade cylinder stock, compounded with not less than 20 per cent. by weight of acidless animal oil, tallow, or tallow oil being preferred.

The compound oil shall show a flashing-point not below 525° F., and a burning-point not below 600°. This test will be made in an open vessel by heating the oil not less than 20° per minute, and applying the test flame every 70°, beginning at 504°.

This oil must flow readily at 60° F., and at a temperature of 350° F., must show a viscosity not lower than that of a pure cane sugar solution containing 52 grams of sugar in 100 cc. of the syrup, the viscosity of the sugar solution being taken at 80° F.

The oil must be transparent, with a reddish-brown or greenish color, free from lumps or specks.

No oil will be accepted which shows more than 5 per cent. of flocculent or tarry matter settled out, after 5 cc. of the oil have been mixed with 95 cc. of 88° gasoline, and allowed to stand for one hour.

Signal Oil

This grade of oil shall be prime white in color, shall contain not less than 40 per cent. by weight of prime lard oil, and shall show a flashing-point not below 200° F., and a burning-point not above 300°. The test will be made in an open vessel by heating the oil not less than 15° per minute and applying the test flame every 7° beginning at 193°

When heated to a temperature of 450° and held there for 5 minutes, the oil must remain clear and transparent, showing but a slight darkening and no separation of flocculent or other matter, either at this temperature or on cooling. The gravity may be from 31° to 34° Beaume.

Oil will not be received which is cloudy from the presence of glue or suspended matter of any kind.

No. 1 *Engine Oil*

This oil shall consist of a high grade of mineral oil, compounded with not less than 10 per cent. by weight of nearly acidless animal oil.

It shall show a gravity of about 29° B., and flashing-point not below 325° F. The test will be made in an open vessel by heating the oil not less than 15° per minute, and applying the test flame once in 7°, beginning at 304°.

Oil received during the months of August and September must have a cold test not above 150° F., and from October 1 to April 1, a cold test not above 5° F., when determined as described below.

From August 1 to April 1 at 80° F., the oil must show a viscosity not lower than that of a pure cane sugar solution containing 80 grams of sugar in 100 cc. of the syrup, and at 150° a viscosity not lower than that of a pure cane sugar solution containing 66 grams of sugar in 100 cc. of the syrup, the viscosity of the sugar solution being taken at 80° F.

From April 1 to August 1, at 80° F., the oil must show a viscosity not lower than that of a pure cane sugar solution containing 88 grams of sugar in 100 cc. of the syrup, and at 150° F. a viscosity not lower than that of a pure cane sugar solution containing 68 grams of sugar in 100 cc. of the syrup, nor higher than that given by a solution of pure cane sugar containing 75 grams of sugar in 100 cc. of the syrup, the viscosity of the sugar solutions being taken at 80° F.

The oil must be transparent with a reddish-brown or greenish color, free from lumps or specks.

No oil will be accepted which shows more than 5 per cent. of flocculent or tarry residue settled out, after five cc. of the oil have been mixed with 95 cubic centimeters of 88° Beaume gasoline and allowed to stand for an hour.

No. 2 *Engine Oil*

The requirements for this oil are identically the same as those for No. 1 engine oil, with the following exceptions:

It must contain not less than 20 per cent. by weight of nearly acidless animal oil.

For October 1 to April 1 the cold test must be not above 10° F., when determined as described below.

Screw Cutting Oil

This oil shall consist of paraffine oil of about 27° B. gravity, compounded with not less than 25 per cent. by weight of fat oil, cotton-seed preferred.

The compounded oil shall show a flashing-point not below 300° F., and a burning point not above 425°. The test will be made in an open vessel by heating the oil not less than 15° per minute, and applying the test flame once in 7°, beginning at 276°.

From October 1 to April 1 the oil must have a cold test not above 15° F., when determined as described below:

> Cold Test—About two ounces of oil are placed in a four-ounce sample bottle, a thermometer inserted and the oil frozen with a mixture of ice and salt. When the oil is hard, the bottle is taken from the freezing mixture, and the frozen oil stirred thoroughly with the thermometer until it will flow. The reading of the thermometer is then taken, and this temperature is regarded as the cold test of the oil.

NOTE.—The viscosity tests will be made upon the Torsion Viscosimeter. Manufacturers not having this instrument, may submit a sample of oil to the Test Department and will be furnished with the information necessary to standardize the instrument they may have in use.

S. T. CHASE MACHINE CO.

Dynamo Oil

(Subject to very high speed, medium pressure, and low temperature.)

1. The specific gravity should be between 0.864 (32° B.) and 0.909 (24° B.) with a preferred gravity of about 0.870 (31° B.)

2. The specific viscosity taken at a temperature of 70° F. should be between 3.50 and 7.50 with a preferred viscosity of about 5.25.

3. The flashing-point (closed tester) must not be less than 330° F., and the burning-point (open tester) should not be below 420° F.

4. Acidity must not exceed 0.10 per cent. of free mineral acid, or more than 1.50 of fatty acid figured as stearic acid.

5. The congealing point must be below 28° F., and the oil must not solidify above 22° F.

6. The evaporation point should not be less than 400° F., and the oil must be absolutely free from gum. The color should be pale yellow or light straw and perfectly transparent.

Samples of one-fourth gallon may be sent for trial analysis, and if found to conform with the specifications will be reported as "accepted" sample; if not up to the requirements they will be marked "rejected."

Every barrel of a shipment must be plainly marked with the gross weight and tare. Parties failing to mark both gross weight and tare must accept the Company's weights without question.

When a shipment of oil is received it will immediately be weighed and where practicable will be emptied and the tare taken. A difference of one per cent. between the invoice and our weights for material adhering to barrel, leakage, variation of scales, etc., will be allowed; if a greater difference exists it will at once be reported for adjustment.

A sample of oil taken at random from any barrel of the shipment will be sent to the laboratory for analysis, and the shipment will be accepted or rejected on this sample.

Definite printed instructions for determining specific gravity, viscosity, acidity, fire test, will be furnished if desired.

In case of dispute, our methods must be used for determining all points set forth in the specifications.

Specifications for Engine Oil for the Department of Docks and Ferries, City of New York

Quality of Engine Oil.—The engine oil to be furnished under this contract shall be one of the following brand or brands equal thereto and approved by the Engineers:

"Vacuum No. 1 Marine Engine Oil."
"Kuhne-Libby Extra Marine Castor Oil."
"Leonard & Ellis Valvoline Engine Oil."
"New York Lubricating Oil Co.'s Diamond B Marengene."
"Fisk Bros.' Luberine Engine Oil;" or equal.

Each can or package of oil delivered under these specifications shall be plainly marked with the marker's name and the brand.

(*a*) Oil must be of the best quality and pass satisfactorily.

(*b*) Specific Gravity.—Must not be less than 0.9000 at a temperature of 60° F.

(*c*) Flashing Point.—Must not be below 420° F.

(*d*) Fire Test.—Must not be less than 475° F.

(*e*) *Freedom from Gumming.*[1]—Just sufficient oil to cover the bottom will be placed in a shallow dish. This will be heated to about 250° F., then cooled slowly. When cold there must be no gummy residue in the oil or in the vessel. Oil must also pass satisfactorily such other tests for gumming as may be ordered by the engineer.

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[1] This gumming is often due to the addition of rope oil,

(*f*) A comomn oil cup holding two ounces will be filled with the oil; two threads of worsted will be used as a wick, and all the oil in the cup must feed through it; the wick not to be touched during the trial. This test to be made at a temperature between 70° and 90° F.

(*g*) Cold Test.—The oil must not solidify at a temperature of 32° F.

(*h*) Freedom from Acid.—A small quantity of oil rubbed on polished brass or copper must not turn the surface of the metal green if allowed to stand for twenty-four hours.

(*i*) Viscosity.—Viscosity at 70° F. must be between 800 and 850 (Engler).

SPECIFICATIONS OF CYLINDER OIL

(*a*) Must be a pure mineral, hydrocarbon oil, with a flash-point of at least 550° F.; burning-point to be above 600° F.; to be free from tarry or suspended matter, acid or alkali, and from mixture or adulteration with animal, vegetable or fish oils, grease, lard or tallow. Specific gravity to be not below 0.900 nor above 0.906 at a temperature of 60° F.

(*b*) A flash-point below 550° F., or the presence of any of the above named adulterations or mixtures, or a gravity different from that specified, will be sufficient to cause the rejection of the oil.

(*c*) Fashing point.—Heat a small quantity of the oil in an open vesssel, not less than 12° per minute, and apply the test flame every 10°, beginning at 250° F.

(*d*) Precipitation test for tarry and suspended matter.—Mix 5 cubic centimeters of oil with 95 cubic centimeters of 88° gasoline, and if there is any precipitation in ten minutes the oil must be rejected.

This test is easiest made by putting 5 cubic centimeters of oil in a 100 cc. graduate, then filling to the mark with gasoline and thoroughly shaking.

(*e*) Volatility.—Keep heated to 400° F. in an open vessel; it must not lose more than 5 per cent. of its weight in two hours.

(*f*) To test for acid or alkali.—It will be sufficient to wash a small quantity of the oil with distilled water, then drain off the water and test it with litmus paper.

Specifications for Lubricating Oil for Dynamo Engines and Other High-Speed Engines Using Forced Lubrication, Issued by the Navy Department, November 17, 1906

(Superseding Specifications 24-O-11 Issued May 25, 1906.)

1. Must be a pure mineral hydrocarbon oil, free from acidity, adulterations, and impurities, with a flash-point (open cup) of at least 350° F.; to be free from saponifiable substances of any character whatsoever; specific gravity to be between 0.865 and 0.875 at 60° F.; to be purchased

and inspected by weight, the number of pounds per gallon to be determined by the specific gravity of the oil at 60° F. multiplied by 8.33 pounds, the weight of a gallon (231 cubic inches) of distilled water at the same temperature.

2. *Viscosity* (by Engler viscosimeter) as compared with distilled water (49) at 90° F.:

At 90° F.	300 to 320
At 150° F.	105 to 115
At 225° F.	65 to 75

3. *Cold Test.*—The oil must flow at a temperature of 32° F.

4. *Freedom from gumming.*—Using a single-wick ½-pint brass oil cup maintained at about 140° F., practically equal quantities of oil must feed through the wick in equal intervals of time for three intervals of eight hours each. At the end of test the wick must be clean and sides of oil cup bright and clean.

INSPECTION AND DELIVERY

5. Before acceptance the oil will be inspected. Samples of each lot will be taken at random, the samples well mixed together in a clean vessel, and the sample for test taken from this mixture. Should the mixture be found to contain any impurities or adulterations, the whole delivery of oil it represents will be rejected, and is to be removed by the contractor at his own expense.

Specifications for Neat's-Foot Oil Issued by the Navy Department

MARCH 24, 1908

(Superseding Specifications 24-O-12 Issued June 22, 1906.)

1. Neat's-foot oil must be free from admixtures of other oils, and must not contain more acidity than the equivalent of 2½ per cent. of oleic acid.

2. It must have a cold test below 10° F., as determined in the following manner: A couple of ounces of the oil will be put in a 4-ounce sample bottle and a thermometer placed in it. The oil will then be frozen, using a freezing mixture of ice and salt if necessary. When the oil has become hard the bottle will be removed from the freezing mixture and the oil allowed to soften, being stirred and thoroughly mixed at the same time by means of the thermometer until the mass will run from one end of the bottle to the other. The reading of the thermometer at this moment will be taken as the cold test of the oil.

Graphite as a Lubricant

Graphite is used either alone or mixed with various oils and greases as a lubricant. Prof. W. F. Goss, of Purdue University, has made a series of extensive experiments upon graphite as a lubricant, and in his report, states:—

"From the earlier and rather limited uses of graphite in lubrication, the field has gradually widened to include its use with light oils, with water, and in some cases, unmixed with other materials. It is no longer regarded merely as a material for an emergency, but now has a place in the ordinary and usual routine of the day."

"Graphite does not behave like oil, but associates itself with one or other of the rubbing surfaces. It enters every crack and pit in the surfaces and fills them, and if they are ill-shaped or irregularly worn, the graphite fills in and overlays until a new surface of more regular outline is produced. When applied to a well-fitted journal the rubbing surfaces are coated with a layer so thin as to appear hardly more than a slight discoloration. If, on the other hand, the parts are poorly fitted, a veneering of graphite of varying thickness, which in the case of a certain experiment was found as great as $^1/_{16}$ inch, will result. The character of this veneering is always the same, dense in structure, capable of resisting enormous pressure, continuous in service without apparent pore or crack, and presenting a superficial finish that is wonderfully smooth and delicate to the touch." From a long and very severe series of experiments, it seems safe to conclude that no journal is likely to be damaged from overheating or kindred causes in the presence of a supply of graphite. It is a fact worthy of all emphasis that, with solid brasses, the presence of flake graphite between the rubbing surfaces makes it impossible to score or otherwise damage the surfaces of the bearings even though the temperature be allowed to run high.

"It goes without saying that the use of graphite, in service which might be rendered without any lubrication whatever, is justified by the more perfect action and the greater durability of the parts affected. Graphite alone, in service for which it is adapted, is to be preferred to oils or greases, because of its superior cleanliness and because of the persistency with which it remains in place after being once applied.

"The experiments with flake graphite as a lubricant justify the following important conclusions:

(*a*) The addition of graphite to oil results in a lower frictional resistance of the journal than would be obtained by the use of oil alone.

(*b*) When graphite is used with oil, the amount of oil required for a given service is reduced.

(*c*) By the use of graphite a light or an inferior quality of oil may be employed for a given service.

(*d*) By the use of graphite, water under favorable conditions may serve as a sufficient lubricant.

(*e*) A small amount of graphite only is required.

(*f*) The supply of too much graphite unduly thickens the oil and correspondingly increases its internal friction due to viscosity.

(*g*) The benefits derived from the use of graphite persist long after its application has ceased. The supply, however, should be constant, though small, for best results."

Even in the cylinders of air compressors there is sufficient moisture to constitute a lubricating film without oil when the surfaces of the cylinders and pistons have been perfected by the presence of graphite.

Lieut. H. C. Dinger, U. S. N. states:—Flake graphite has the peculiar properties of not being affected, either chemically or physically, by any temperature encountered in a cylinder. It is not easily carried away from the wearing surfaces, can stand any pressure, and requires only an infinitestimal clearance space between surfaces by filling up all the minute cavities and irregularities in the surfaces, giving in a short time, a beautiful, hard-polished surface which requires relatively little lubricant.

Specifications for Lubricating Graphite Issued by the Navy Department

JANUARY 17, 1907

1. It may be of the flake or amorphous variety. Amorphous graphite must be ground fine enough to pass through a No. 20 bolting cloth.

2. Samples taken from any lot must show upon analysis at least 85

per cent. of pure graphite. It must be free from grit, dirt, or any other deleterious substance.

3. It must be put up in air-tight rectangular tin cans with screwed tops, each containing 1 or 5 pounds, as may be required.

4. Each can must be marked with the name of the material, the trademark, if any, and the name of the manufacturer.

Remarks on Lubricating Oils

Lubricating oils and lubricants in general may be divided into the following classes, viz.:

Cylinder oils, dynamo oils, engine oils, machine oils, greases.

The conditions usually met with in the lubrication of machinery may be stated as follows:

Pressure, heavy, medium, or light.

Speed, fast, medium, slow.

Temperature, high, medium, low.

Cylinder oils should be sub-divided into two classes, one for high steam pressure with an extreme temperature, and one for medium pressure and temperature. With the steam gauge at an average above 90 lbs. a cylinder oil with a high fire test (600° F. or above) should be used.

With a heavy pressure on the working part of the cylinder it is essential that the specific gravity of the oil be not less than 0.885.

The congealing point in cylinder oils is of minor importance, yet it should not be too high; a common average is from 40° to 50° F.

Viscosity is a very important feature and must be carefully considered. An oil with a viscosity of about 3.50 at 180° F. (water being rated as 1.00 viscosity) will feed through the oil cup at the rate of three to four drops per minute, while an oil with a viscosity of 5.00 will feed only half as fast. When once in the cylinder and in contact with the high temperature the viscosity is reduced to its minimum.

Acidity, or free acid is a detrimental quality. Most cylinder oils contain tallow or animal oil in conjunction with the mineral oil, animal oils become rancid under certain conditions and are partly converted into stearic and oleic acids which have a decided action on metals. The free acid in animal oils and greases is called "free fatty acid." Mineral oil should be free from acid, but in some cases where the oil has not properly been treated it contains traces of sulphuric or other mineral acids.

The lower the free acid, the better the oil. It may be stated that 3.00 per cent. of fatty acid or 0.50 per cent. of mineral acid is the danger point.

For good results the acid should be less than half this amount.

Dynamo oils—are generally used in machines running at a high rate of speed.

With a close fitting journal and fast speed a thin oil should be used, but for medium speed and a loose journal a thick or high viscosity oil gives better results.

A dynamo oil with a viscosity of 4.50 to 6.50 at 70° F., is considered a thin oil, and one with a viscosity of 9.00 to 11.00 a thick oil.

If there is heavy pressure on the bearings, then use an oil with a high specific gravity.

High speed machines should always be run with a flooded bearing, *i. e.,* excess of oil, as it is imperative that the temperature be kept down.

Low fire-test oils are the rule in this class of work, but no oil with a fire-test under 300° F. should be used.

The congealing point is of little importance unless the oil is to be stored or used in a very cold room, in which event the cold test must necessarily be low. The free acid in a dynamo oil should be, and generally is less than 0.50 per cent., in most cases there is no acid whatever.

Engine and Machine Oils

For tight--fitting bearings a high specific gravity and low viscosity oil is required.

For heavy pressure and loose-fitting bearings a high specific gravity and a thick or high viscosity oil should be used.

The general temperature of the machine will specify the fire-test of the oil to be used. The average fire-test for engine, dynamo and machine oils varies from 350° F. to 500° F.

The congealing point should be regulated according to the conditions of temperature; thus, if the machinery is located in a place where the temperature is occasionally below freezing (32° F.) the oil should have a low cold test.

Lard oil and several other animal oils congeal at about 36° F., while mineral oils are generally below 22° F.

Briefly stated, the physical properties of an oil may be said to comply with the following conditions:

For heavy pressure, a high specific gravity.

For tight journals, a high specific gravity.

Fast speed (tight journals) use a low viscosity oil.

Fast speed (loose journals) use a high viscosity oil.

High temperatures call for a high fire-test.

Low temperature, for a low cold test.

Line machinery needs an oil free from gum and with little free acid.

Heavy, slow-moving machines, require thick oils or greases for their lubrication.

The Calorific Power of Petroleum Oils and the Relation of Density to Calorific Power

The purpose of this paper is to put on record the calorific power of a considerable number of representative American petroleum oils and to point out an approximate relationship between the density and the calorific power of such oils.[1]

While among the homologues of a given series of hydrocarbons, decreasing proportions of hydrogen might be assumed to involve an increase in density and decrease in heat of combustion or calorific power, it would not necessarily follow that such a relation would obtain for the mixtures of hydrocarbons which constitute the crude petroleums or their commercial products. Nor have we been able to find in the literature sufficient comparable data to give even an approximate expression of the quantitative relation to be expected between density and the calorific power of petroleum products.

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[1] A. C. Sherman and A. H. Kropff, *Jour. Amer. Chem. Soc.* Oct., 1908, pp. 1626-31.

DENSITIES AND HEATS OF COMBUSTION OBSERVED AND CALCULATED.

No.	Specific gravity 15°/15°.	Baumé degrees.	Calories per kilogram.	B. T. U. per pound.	B. T. U. calculated.	Percentage error.	Description.
1	0.7100	67.2	11,733	21,120	20,938	—0.91	Gasoline
2	0.7175	65.1	11,327	20,389	20,854	+2.33	"
3	0.7200	64.4	11,404	20,527	20,726	+0.99	"
4	0.7709	51.6	11,132	20,038	20,314	+1.38	[1]
5	0.7830	48.8	11,121	20,018	20,206	+0.92	Kerosene
6	0.7850	48.35	11,119	20,014	20,194	+0.89	California refined
7	0.7945	46.2	11,128	20,030	50,098	+0.33	West Va. crude
8	0.7950	46.1	11,186	20,135	20,094	-0.20	Kerosene
9	0.7964	45.8	11,242	20,236	20,082	+0.70	[1]
10	0.8048	44.0	11,149	20,068	20,010	—0.29	Ohio crude
11	0.8059	43.7	11,143	20,057	19,998	—0.29	Penna. crude
12	0.8080	43.2	11,001	19,802	19,979	+0.88	California refined
13	0.8103	42.8	11,090	19,963	19,962	±0.00	Kansas refined
14	0.8237	40.0	10,981	19,766	19,850	+0.42	West Va. crude
15	0.8248	39.7	11,015	19,827	19,838	+0.05	California refined
16	0.8261	39.5	11,123	20,021	19,830	—0.95	West Va. crude
17	0.8321	38.2	10,972	19,757	19,778	+0.11	[1]
18	0.8324	38.2	10,990	19,782	19,778	—0.02	Penna. crude
19	0.8418	36.3	10,950	19,710	19,702	—0.04	Ohio crude
20	0.8421	36.25	10,997	19,795	19,698	—0.48	Indian Territory
21	0.8536	36.0	11,069	19,924	19,690	—1.17	[1]
22	0.8466	35.4	10,936	19,685	19,666	—0.09	Indian Territory
23	0.8500	34.7	10,953	19,715	19,638	—0.38	California refined
24	0.8510	34.5	10,958	19,724	19,630	—0.47	Kansas crude
25	0.8514	34.45	10,945	19.701	19,630	—0.35	[1]
26	0.8534	34.05	10,991	19,784	19,610	—0.86	[1]
27	0.8580	33.2	10,772	19,389	19.578	+0.95	Kansas crude
28	0.8597	32.8	10,766	19,379	19,562	+0.95	Illinois crude
29	0.8616	32.5	10,967	19,741	19 550	—0.95	[1]
30	0.8640	32.05	10,867	19,555	19,530	—0.12	California refined
31	0.8648	31.9	10,920	19,656	19,526	—0.65	Penna. feul oil
32	0.8660	31.65	10,864	19,555	19,516	—0.19	Fuel oil
33	0.8670	31.5	10,850	19,530	19,510	—0.10	Penna. fuel oil
34	0.8690	31.1	10,852	19,534	19,494	—0.20	Indian Territory
35	0.8708	30.8	10,919	19,654	19,482	—0.86	[1]
36	0.8712	30.7	10,879	19,614	19,478	--0.68	[1]
37	0.8745	30.1	10,752	19,354	19,454	+0.50	Kansas crude
38	0.8773	29.6	10,794	19,429	19,434	+0.03	Penna. fuel oil
39	0.8800	29.0	10,804	19,447	19,410	—0.18	Kansas crude
40	0.8807	29.0	10,797	19,435	10,410	—0.47	[1]
41	0.8810	28.9	10,797	19,435	19,406	—0.15	
42	0.8820	28.75	10,913	19,643	19,400	—1.22	[1]

Densities and Heats of Combustion Obtained and Calculated—(*Continued*).

No.	Specific gravity 15°/15°.	Baumé degrees.	Calories per kilogram.	B. T. U. per pound.	B. T. U. calculated.	Percentage error.	Description.
43	0.8828	28.7	10,694	19,249	19,396	+0.73	Kansas crude
44	0.8833	28.5	10,819	19,474	19,390	—0.42	[1]
45	0.8860	28.0	10,808	19,454	19,370	—0.42	Indian Territory
46	0.8862	28.0	10,762	19,372	19,370	—0.01	[1]
47	0.8900	27.3	10,788	19,418	19,342	—0.39	Indian Territory
48	0.8914	27.1	10,690	19,242	19,332	+0.45	Texas crude
49	0.8970	26.1	10,753	19,355	19,294	—0.31	
50	0.9007	25.4	10,755	19,359	19,267	—0.47	[1]
51	0.9050	24.7	10,682	19,228	19,238	+0.05	
52	0.9065	24.45	10,751	19,352	19,228	—0.63	[1]
53	0.9066	24.4	10,605	19,089	19,226	+0.69	Kansas crude
54	0.9087	24.1	10,712	19,282	19,213	—0.35	
55	0.9114	23.6	10,724	19,303	19,194	—0.55	Kansas crude
56	0.9137	23.2	10,571	19,028	19,178	+0.76	Texas crude
57	0.9153	22.95	10,692	19,246	19,168	—0.39	Texas crude
58	0.9155	22.9	10,560	19,008	19,166	+0.80	Texas crude
59	0.9158	22.9	10,318	18,572	19,166	+2.58	California crude
60	0.9170	22.7	10,613	19,103	19,157	+0.28	Fuel oil
61	0.9179	22.5	10,433	18,779	19,150	+1.94	California crude
62	0.9182	22.5	10,547	18,985	19,149	+0.83	California crude
63	0.9336	20.0	10,600	19,080	19,048	—0.16	Texas crude
64	0.9644	15.2	10,327	18,589	18,858	+1.42	California crude

[1] Obtained by fractional distillation of commercial fuel or gas oils ; Nos. 4, 25, 36, 44 ; and 52 were the successive fifths from one sample ; Nos. 9, 21, 26, 29, and 42 from a second Nos. 17, 35, 40, 46 and 50 from a third.

Fuel-Oil Specifications

General specifications for the purchase of fuel oil are given as follows:

In determining the award of a contract, consideration will be given to the quality of the fuel offered by the bidders, as well as the price, and should it appear to be to the best interest of the Government to award a contract at a higher price than that named in the lowest bid or bids received, the contract will be so awarded.

Fuel oil should be either a natural homogeneous oil or a homogeneous residue from a natural oil; if the latter, all constituents having a low flash point should have been removed by distillation; it should not be composed of a light oil and a heavy residue mixed in such proportions as to give the density desired.

It should not have been distilled at a temperature high enough to burn it, nor at a temperature so high that flecks of carbonaceous matter began to separate.

It should not flash below 60° C. (140° F.) in a closed Abel-Pensky or Pensky-Martens tester.

Its specific gravity should range from 0.85 to 0.96 at 15° C. (59° F.); the oil should be rejected if its specific gravity is above 0.97 at that temperature.

It should be mobile, free from solid or semi-solid bodies, and should flow readily, at ordinary atmospheric temperatures and under a head of 1 foot of oil through a 4-inch pipe 10 feet in length.

It should not congeal nor become too sluggish to flow at 0° C. (32° F.).

It should have a calorific value of not less than 10,000 calories per gram (18,000 British thermal units per pound); 10,250 calories to be the standard. A bonus is to be paid or a penalty deducted according to the method stated under Section 21, as the fuel oil delivered is above or below this standard.

It should be rejected if it contains more than 2 per cent. water.

It should be rejected if it contains more than 1 per cent. sulphur.

It should not contain more than a trace of sand, clay or dirt.

Each bidder must submit an accurate statement regarding the fuel oil he proposes to furnish. This statement should show: The commercial name of the oil; the name or designation of the field from which the oil is obtained; whether the oil is a crude oil, a refinery residue, or a distillate; the name and the location of the refinery, if the oil has been refined at all.

The fuel oil is to be delivered f. o. b. cars or vessel, according to the manner of shipment, at such places, at such times, and in such quantities as may be required.

Should the contractor, for any reason, fail to comply with a written order to make delivery, the Government is to be at liberty to buy oil in the open market and charge against the contractor any excess of price, above the contract price, of the fuel oil so purchased.

The Analysis of Lubricating Oils Containing Blown Rape-Seed and Blown Cotton-Seed Oils

Rape-seed oil has long been the standard oil in Europe for lubrication. Its constancy of viscosity at varying temperatures, its non-liability to acidity as compared with other seed oils, and its low cold test, unite in producing the results required of a good lubricant. It, however, is no exception to the rule that vegetable and animal oils suffer partial decomposition when subjected to high temperature produced by friction, with the result that fatty acids are liberated and corrosion of bearings produced.

The substitution of mineral oils in varying proportions with rape-seed oil has reduced this tendency, this reduction being determined by the percentage of mineral oil present, as the latter liberates no free acids.

It is a peculiar fact, however, that a mineral oil alone does not give as satisfactory results in lubrication (especially cylinder lubrication[1]) as does a mixture of mineral and vegetable or mineral and animal oils, one of the primary causes being that the viscosity of mineral oils rapidly diminishes at high temperatures, whereas the reduction of viscosity of vegetable and animal oils is very much less.

If it were not for this peculiarity between these two classes of oils, mineral lubricating oils could easily supplant (on the score of cheapness) all other oils used in lubrication.

The admixture of oils then being required for the better class of lubricants, it follows that in England where rape-seed oil has been the standard, its use should be continued in compounded oils.

The proportion of rape-seed oil added to mineral oil varies from 5 to 20 per cent. Where the mineral oil is a clear paraffin oil 20 per cent. of the seed oil is used; where the mineral oil is a dark, heavy oil, 5 per cent. is generally added.

The separation and estimation of the rape-seed oil in these mixtures presents no difficulty to the analytical chemist when no other seed oil is present, since the saponification of the seed oil, the separation of the fatty acids and recognition of the same are a part of the usual chemical work of this character. The recognition of the constituents of a mixed lubricating oil by analysis is a very different problem from giving a formula by which the mixture can be made. This is evidenced as follows:

---

[1] *The Railroad and Engineering Journal*, 64, 73-126.

Suppose the analysis shows—

	Per cent.
Rape-seed oil	20
Paraffine oil	80

Paraffin oil varies in specific gravity from 0.875 to 0.921, and it is essential to include in the report of the analysis not only the amount of paraffin oil, but also the gravity, since paraffin oil of gravity 0.875 is a very different product from that of 0.921 gravity, the former selling at 7½ cents and the latter at 23 cents per gallon. This determination can be made by taking the gravity of the original mixed oil (0.912), then knowing by the analysis that 20 per cent. is rape-seed oil (gravity 0.918), the gravity of the 80 per cent. of paraffin oil is easily calculated. Thus:

x = specific gravity of rape-seed oil (0.918),
v = specific gravity of paraffin oil,

Then

$$\begin{aligned} \tfrac{1}{5}x + \tfrac{4}{5}y &= 0.912, \\ 0.183 + \tfrac{4}{5}y &= 0.912, \\ \tfrac{4}{5}y &= 0.729, \\ y &= 0.910. \end{aligned}$$

The mixture being composed, therefore, of—

	Per cent.
Paraffin oil (sp. gr. 0.910)	80
Rape-seed oil (sp. gr. 0.918)	20

The direct determination by analysis from the ether solution of the mineral oil in the mixture does not give an oil of the same specific gravity as the mineral had before it was mixed with the seed oil. This can be accounted for by the volatilization of a portion of the lighter hydrocarbons of the mineral oil when the ether is expelled during the analysis. For this reason the determination of the percentage of seed oil and the calculation of the mineral oil offers less liability to failure than finding the mineral oil directly.

The introduction of blown rape-seed oil instead of the normal rape-seed oil complicates the investigation and renders the use of the formula above given valueless. Rape-seed oil has a gravity of 0.915 to 0.920. Rape-seed oil blown has a gravity of from 0.930 to 0.960.

Two difficulties are immediately presented: (1) The chemical analysis does not indicate whether the rape-seed oil is blown or not; (2) The use of the formula given without the correct gravity of the blown oil would give false results regarding the paraffin oil. To overcome this difficulty some synthetical work is required.

Suppose the specific gravity of the mixed oil is 0.922 and the analysis shows 20 per cent. of rape-seed oil. It will be necessary then to produce a mixture in these proportions that will duplicate the original sample. A check upon this will be the viscosity of the original sample as compared with the one to be made by formula. Thus:

The original oil has a gravity of 0.922, contains (by analysis) 20 per cent. of rape-seed oil, and has a viscosity at 100° F. of 335 seconds (Pennsylvania Railroad pipette).

First.—Make a mixture of paraffin oil (sp. gr. 0.910) generally used in this character of lubricant, 80 per cent., and rape-seed oil (unblown), 20 per cent. The viscosity is 165 seconds, showing that this mixture cannot be used in place of the original oil.

Second.—Make a mixture of paraffin oil (sp. gr. 0.910) and rape-seed oil partially blown (sp. gr. 0.930), in the same proportions as above. The resulting viscosity is 267 seconds, showing that the compound is still lacking in viscosity.

Third.—Make a mixture of paraffin oil (sp. gr. 0.910), eighty parts, and rape-seed oil, (blown) sp. gr. 0.969, twenty parts; the viscosity is 332 seconds.

This now fulfills the conditions required and the synthetical sample agrees with the original in gravity, composition, and viscosity.

The use of blown rape-seed oil is being gradually replaced by blown cotton-seed oil. The latter, which has had but a limited use in lubrication, owing to its liability to acidity, has been greatly improved by this process of "blowing," which is nearly complete oxidation of the oil under comparatively high temperature.

This largely prevents the occurrence of the acidity in the oil, and thus the main objection to its use in lubrication disappears. It is much cheaper than rape-seed oil, since it costs 30 cents per gallon, to 60 cents per gallon for the latter. The chemical reactions of the two oils are very similar, and careful analytical work is required that the chemist be not misled.

The following table of comparisons will indicate this:

Specific Gravity

Cotton-seed oil	0.920 to 0.925
Rape-seed oil	0.915 to 0.920
Blown cotton-seed oil	0.930 to 0.960
Blown rape-seed oil	0.930 to 0.960

Viscosity (Pennsylvania Railroad Pipette) at 100° F.

	Seconds
Cotton-seed oil (sp. gr. 0.925)	162
Rape-seed oil (sp. gr. 0.918)	210
Blown cotton-seed oil (sp. gr. 0.960)	2,143
Blown rape-seed oil (sp. gr. 0.960)	2,160

Heidenreich's Test

	Before stirring	After stirring
Cotton-seed oil	Faint reddish brown	Brown
Rape-seed oil	Yellow-brown	Brown

Massie's Test

Cotton-seed oil	Orange-red
Rape-seed oil	Orange

Iodine Absorption

Cotton-seed oil	104 to 114
Blown cotton-seed oil	93 to 103
Rape-seed oil	102 to 108
Blown rape-seed oil	94 to 100

In the comparison of the two oils, when not mixed with a mineral oil, the above tests can be used. The conditions are altered, however, when either one or both are so mixed, since these tests apply only to the pure oils and not to those reduced with large percentages of mineral oil. After the separation of the seed oil from the mineral oil by saponification the identification of the seed oil depends upon the reactions of the fatty acids obtained, and a careful examination and comparison of these reactions shows that the melting-points have the greatest difference and thus become a means of recognition.

Thus, the fatty acids from rape-seed oil melt at 20° C., and from cotton-seed oil at 30° C. Hence, if upon analysis of a lubricating oil under above conditions, the fatty acids obtained show a melting-point of 20° C. the seed oil can be pronounced rape-seed oil.

If the melting-point is between these limits, say 23° C., the seed oils are present in a mixture, the proportions of which can be determined by the following formula:

w_1 = proportion of rape-seed oil,
w_2 = proportion of cotton-seed oil,
w_3 = weight of mixture (20 per cent.),
t_1 = temperature of melting-point of fatty acids of rape-seed oil,
t_2 = temperature of melting-point of fatty acids of cotton-seed oil,
t_3 = temperature of melting-point of mixed fatty acids.

Then

$$w_1 = w_3 \frac{t_3 - t_2}{t_1 - t_2}$$

$$w_2 = w_2 \frac{t_3 - t_1}{t_2 - t_1}.$$

Inserting the value:

$$w_1 = 20 \frac{23 - 30}{20 - 30} = 14 \text{ per cent.}$$

$$w_2 = 20 \frac{23 - 20}{30 - 20} = 6 \text{ per cent.}$$

Or,

	Per cent.
Paraffin oil	80
Rape-seed oil	14
Cotton-seed oil	6
Total	100

By synthetical work upon these proportions, with comparison of viscosities of the sample submitted with the product, the result will be not only a correct analysis but a working formula can be given by which a manufacturer can duplicate the original oil.

The Analysis of Cylinder Deposits

The deposits in steam cylinders, formed by the decomposition of lubricating oils, may be classed as simple or compound, depending upon whether the deposit is due to the decomposition of the oil alone or if foreign matters, carried over in the steam from the boilers, are also present.

In the former case, carbon, hydrocarbons, oils, and iron oxide are the principal constituents, whereas, in the latter, oleate of lime, carbonate of lime, and silica are often present in addition to the former.

The following analysis of a sample from a locomotive cylinder would indicate a simple deposit:

		Per cent.
Moisture		2.28
Oils soluble in ether	animal	10.54
	mineral	11.23
Hydrocarbons insoluble in ether		47.97
Fixed carbon		23.73
FeO		2.83
Undetermined		1.42
Total		100.00

And the one given below, of a deposit from the steam cylinders of a large stationary engine, would show that scale-forming material from the boilers had become a component.

		Per cent.
Moisture		13.12
Oils soluble in ether	animal	8.15
	mineral	7.86
Soap		2.10
Hydrocarbons insoluble in ether		1.67
Fixed carbon		2.71
Oxides of iron and aluminum		6.81
SiO_2		3.65
$CaCO_3$		43.22
$MgCO_3$		10.17
Undetermined		0.44
Total		100.00

In many samples I have found copper and zinc in the deposits, formed by the corrosive action of the liberated oleic acid from the animal oil upon the brass or composition bearings.

This corrosive action is very marked where a poor quality of lubricating oil, composed of animal or vegetable oil, is used, whereas, a pure neutral mineral oil has no acid action at steam temperature. Oftentimes the statement has been made to me, when the deposit was given for analysis, "All of our lubricating oil is pure mineral oil; we use no other." And yet, upon analysis, lard oil would be shown in comparatively large amounts.

This is accounted for from the fact that while the consumer believes he is using pure mineral oil—which was sold to him as such—the manufacturer has introduced from 5 to 30 per cent. of lard or cotton-seed oil.

A large majority of the so-called "pure mineral" lubricating oils for cylinder use contain at least 5 per cent. of animal oil; and it is the exception and not the rule to find a "pure mineral" oil for cylinder lubricating purposes.

An analysis of a deposit from the steam cylinder of a large freight steamer gave as a result:

		Per cent.
Moisture		16.16
Oils soluble in ether	castor oil	26.19
	mineral	32.50
Fixed carbon		7.92
CuO		0.50
FeO		15.10
Undetermined		1.63
Total		100.00

Pure mineral lubricating oil was supposed by the officers of the vessel to be the only lubricant used, and special care had been taken to secure it, but it appears that the engineer added a small amount of castor oil to the mineral oil, as, in his opinion, it made a better lubricant.

The decomposition of the castor oil and liberation of the fatty acids was the primary cause of the deposit.

The action of the fatty acids upon the iron and metal bearings results in different products. That is to say, while the copper when present has generally been estimated as copper oxide the iron may exist as oxide or as metallic iron or both.

No doubt the oleic acid acts to form salts of these metals, but it is certain, in many instances, that when formed, they are immediately decomposed or partially so, and a resulting mixture formed that is somewhat difficult of analysis.

In the analysis here given, it will be noticed that the iron was found both as metal and as oxide.

		Per cent.
Moisture		3.77
Oils soluble in ether	animal	21.27
	mineral	19.60
Soap		traces
Fixed carbon		10.90
FeO		14.01
Fe		27.85
PbO		0.82
CuO		1.07
Undetermined		0.71
Total		100.00

The evolution of hydrogen by hydrochloric acid, from the deposit, after all the oils and fatty substances had been removed, indicated the presence of metallic iron, and the analysis of the residue, after the combustion of the fixed carbon, gave figures by which the ratio of iron and iron oxide could be determined. A portion of the deposit, after extraction of oils by ether,[1] is dried, then weighed, the hydrocarbons driven off by heat, and the amount of fixed carbon present converted by combustion with sulphuric acid and chromium trioxide into carbon dioxide and weighed, this weight being calculated back to carbon.

Another portion of the same residue is ignited in a platinum crucible until the carbon is all consumed, then weighed. If the amount of carbon found is small and iron large, this weight may be larger than the original weight of the residue taken, owing to oxidation of metallic iron to ferric oxide.

Knowing the weight of carbon, and by making a determination of iron in another sample before ignition, the amount of iron oxide is easily found.

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[1] Soxhlet apparatus as shown in Fig. 153 is well adapted for this purpose.

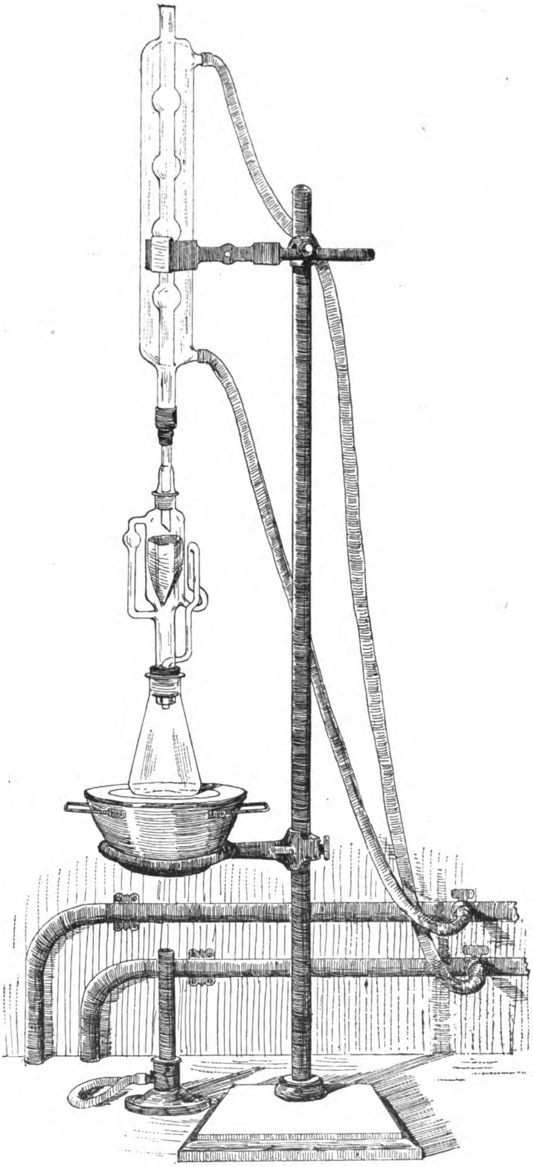

Fig. 153.

SCHEME FOR THE ANALYSIS OF CYLINDER DEPOSITS

<table>
<tr><td colspan="9">(1) Weigh out 10 grams of the deposit upon a tared filter; dry at 102° C. to constant weight. Loss is moisture. The filter and contents are transferred to a Soxhlet tube (Fig. 153) and exhausted with ether.</td></tr>
<tr><td colspan="7">(2) Residue.—If qualitative analysis has shown soap to be present, the residue is dried slowly and then water poured upon the filter until extraction is complete.</td><td colspan="2" rowspan="2">(3) Solution.—Contains the free oils. Determine their weight by evaporation of the ether; then treat the residue with alcoholic solution of potash. Drive off the alcohol, treat the mass with warm water, transfer to a separatory funnel, allow to cool, add sufficient ether, agitate, and set aside six hours.
Draw off the soap solution.</td></tr>
<tr><td colspan="6">(4) Residue.—Dry; take a weighed portion; transfer to a weighed covered platinum crucible, and ignite gently; air excluded. The loss represents volatile hydrocarbons insoluble in ether. Determine this, then ignite at lowest possible heat until weight is constant. (Accurate determination of fixed carbon requires oxidation with $H_2SO_4 + CrO_3$ to CO_2 and weighing as such, first treating with dilute HCl to drive off any carbonic acid united with CaO or MgO.) Loss is fixed carbon. Residue contains the oxides of the metals, etc. The residue is weighed, then transferred to a porcelain capsule, HCl added with a few drops of HNO_3, and evaporated to dryness. Take up with HCl and water, filter; wash well.</td><td rowspan="6">(5) Solution.—Evaporate to dryness and determine weight of soap. Separate the fatty acids and test qualitatively for animal and vegetable oils.
CO_2 is determined by taking a weighed sample of residue marked (4) treating with HCl and weighing the CO_2 evolved.</td></tr>
<tr><td rowspan="5">(6) Residue.—Insoluble mineral matter. SiO_2, etc.</td><td colspan="5">(7) Solution.—Pass H_2S; filter.</td><td rowspan="5">Soap Solution.—Warm gently for one-half hour, acidify with dilute H_2SO_4, allow separated fatty acids to collect and weigh with wax. Consult <i>J. Anal. Appl. Chem.</i>, 5, 710.</td><td rowspan="5">Ether Solution. — Evaporate to dryness and determine weight of mineral oil. Test also for soaps insoluble in water, but soluble in ether. (Lead soap, zinc soap, lime soap, etc.</td></tr>
<tr><td rowspan="4">(8) Residue.—Sulphides of copper and lead. Separate and determine their amounts and calculate to oxides of copper and lead.</td><td colspan="4">(9) Solution.—Boil out H_2S, oxidize with HNO_3, add ammonia to alkaline reaction; filter.</td></tr>
<tr><td rowspan="3">(10) Residue.—Fe_2O_3 (Al_2O_3). Dry, ignite, and weigh as such, and calculate to FeO. If qualitative analysis has shown iron, as metal, determine the Fe also.</td><td colspan="3">(11) Solution.—Add H_2S to solution and filter.</td></tr>
<tr><td rowspan="2">(12) Residue. ZnS.—Dissolve in HCl and determine zinc as carbonate and calculate to zinc oxide.</td><td colspan="2">(13) Solution.—Boil out H_2S, add ammonium oxalate and a few drops of ammonium hydroxide; set aside six hours; filter.</td></tr>
<tr><td>(14) Residue, CaO.—Ignite and weigh as CaO.</td><td>(15) Solution.—Add solution sodium phosphate; set aside 2 hours; filter, dry, ignite and weigh as $Mg_2P_2O_7$. Calculate to MgO.</td></tr>
<tr><td>SiO_2.</td><td>CuO. PbO.</td><td>Fe, FeO, and (Al_2O_3).</td><td>Zno.</td><td>CaO.</td><td>MgO.</td><td>CO_2.</td><td>Animal oil</td><td>Mineral oil.</td></tr>
</table>

Where quantitative analysis has shown the deposit to be a simple one, the analysis can be stated as follows:

		Per cent.
Oils soluble in ether	animal	——
	mineral	——
Moisture		——
Hydrocarbons		——
Fixed carbon		——
FeO		——
Fe		——
Total		——

For a complex deposit, the following form can be used:

		Per cent.
Moisture		——
Oils soluble in ether	animal	——
	mineral	——
Soap		——
Hydrocarbons insoluble in ether		——
Fixed carbon		——
Fe		——
FeO		——
CuO		——
PbO		——
ZnO		——
CaO		——
MgO		——
CO_2		——
SO_3		——
SiO_2, etc		——
Total		——

Where the lime and magnesia exist in amounts more than necessary to combine with the carbon dioxide and sulphur trioxide present, the excess may have united with oleic acid to form soaps insoluble in water, but soluble in ether.

In some instances the lead oxide and zinc oxide will be found only in the ether soap solution (3), as lead and zinc oleates, but in others, while they undoubtedly first existed as oleates, they had become decomposed, and the lead and zinc oxides would be found in section (8) of the above scheme.

The following is an analysis of a cylinder deposit, "Mica Grease" having been used as a lubricant:

		Per cent.
Moisture		11.23
Oils soluble in ether	animal	9.05
	mineral	6.28
Soap (CaO + MgO united with fatty acids)		43.90
Fixed carbon		6.33
Oxides of iron and aluminum		6.59
CaO		3.15
MgO		2.19
CO_2		6.27
Silica aud mica		5.01
Total		100.00

Technical Examination of Petroleum

This is usually performed by fractional distillation of the petroleum into three classes of distillates.

1. Light oils, distilling over up to 150° C.
2. Illuminating oils distilling over from 150° C. to 300° C.
3. Residuum.

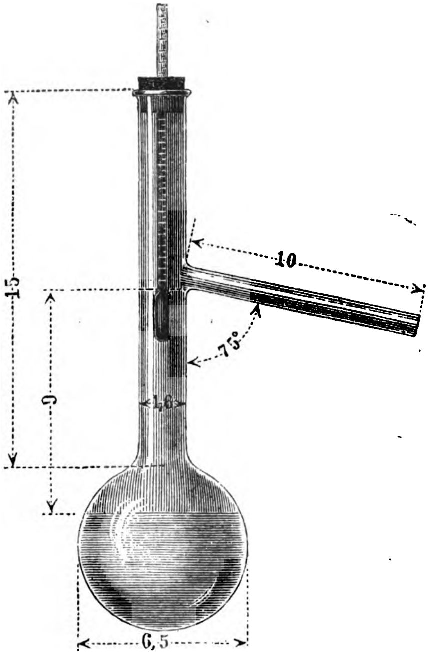

Fig. 116.

The method of Engler, which is largely used for this purpose, requires a glass flask of the form shown in Fig. 116. The measurements given in the figure are stated in centimeters. The flask is connected with a condenser in the usual manner.

One hundred cc. of the oil are taken and the temperature in the flask is regulated that 2.5 cc. of the distillate pass over every minute. Chemists vary the method of distillation, some using 300 cc. of the oil and a larger flask of same form, though without standard rules respecting the number of distillates to be obtained: thus A. Bourgougnon and J. Mandel[1] report the analysis of a sample of Ohio petroleum in which the distillation was in 50 parts, each part representing 2 per cent. by volume, the distillation commencing at 23° C. The composition of the oil being given as 16 per cent. of naphtha, 70° B., 68 per cent. kerosene, 6 per cent. of paraffin oil, and 10 per cent. of residuum. Durand Woodman[2] gives an analysis of a crude petroleum from Ohio. Three hundred cc. of the oil were taken and 18 distillates each of 15 cc. (5 per cent. of total) were obtained. The results in detail were as follows:

Number of distillate	F° Fahr.	B° Baumé	Per cent.
1	160	70.5	5
2	200	65.0	10
3	210	61.0	15
4	250	57.5	20
5	263	54.0	25
6	277	52.0	30
7	348	48.0	35
8	354	45.0	40
9	370	43.0	45
10	400	41.0	50
11	427	40.0	55
12	...	40.0	60
13	476	39.0	65
14	486	40.0	70
15	490	39.0	75
16	486	40.0	80
17	466	41.0	85
18	450	41.0	90
Residuum	...	....	100

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[1] *J. Amer. Chem. Soc.*, 13, 168.

[1] *Ibid.*, 13, 180.

The result being:

	Per cent.
Naphtha	10
Illuminating oil	50
Lubricating oil	30
Residuum	10
Total	100

A distillation of a Mexican petroleum, by the writer, made by the Engler method, gave:

	Per cent.
Naphtha	10.0
Illuminating oil	60.0
Lubricating oil	15.5
Tar and residuum	14.5
Total	100.0

Another sample of the same oil, submitted to a somewhat higher temperature during the distillation, using a similar flask excepting that the delivery tube was 1.5 inches higher in the neck of the flask (requiring higher heat upon the petroleum for the same distillates as in the former case), gave a lower percentage of heavy oils, and a higher percentage in illuminating oils, the result being:

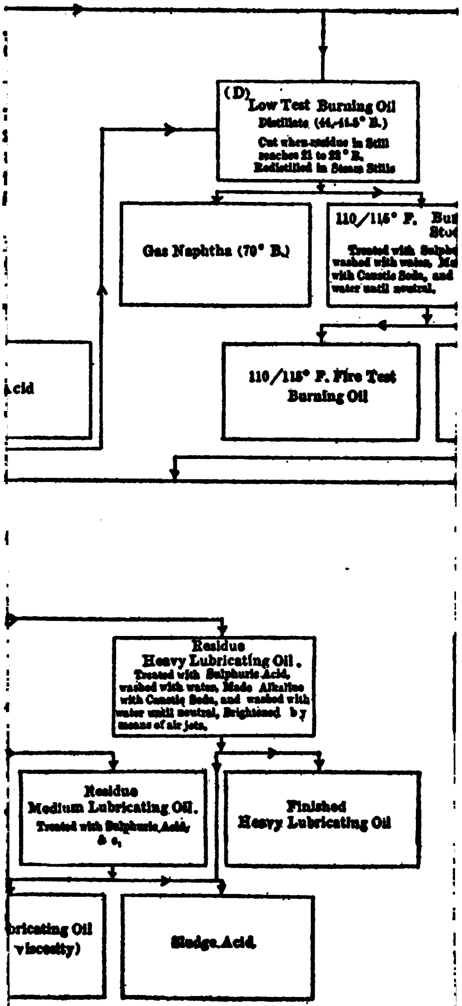
	Per cent.
Naphtha	11.0
Illuminating oil	64.0
Lubricating oil	10.3
Residuum	14.7
Total	100.0

By a careful regulation of the heat, the amount of illuminating oil can be increased or decreased to a certain percentage as desired.

The following table, which is taken from the *Petroleum Review* of August 15, 1908, gives a good idea of the approximate composition of the crude oils from the different oil fields in the United States:

APPROXIMATE COMPOSITION OF THE CRUDE OILS OF THE UNITED STATES.

Products.	Appalachian Per cent.	Lima-Ind. Per cent.	Mid-Cont. Per cent.	Gulf Per cent.	Cal. Per cent.	Corsicana Per cent.	Colo. Per cent.
Naphthas — gasolene, benzine, etc.	12	11½	11	3	6	7	3½
Illuminating oil	67	43	41	15	18	50	35
Lubricating oils (including grease)	12½	15	—	6	1½	—	3
Gas oil	4		25	45	72	—	—
Fuel oil (including acid oil, asphaltum oil and asphalt)		25	20	28		40	55
Paraffin wax	2	2	—	—	—	—	—
Total	97½	96½	97	97	97½	97	96½



EN DISTILLED DESTRUCTIVELY.*

Comparison of Centigrade and Fahrenheit Degrees

Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.
2500	4532	278	532.4	228	442.4
2000	3632	277	530.6	227	440.6
1500	2732	276	528.8	226	438.8
1200	2192	**275**	**527**	**225**	**437**
1000	**1832**	274	525.2	224	435.2
950	1742	273	523.4	223	433.4
900	1652	272	521.6	222	431.6
850	1562	271	519.8	221	429.8
825	1517	**270**	**518**	**220**	**428**
800	**1472**	269	516.2	219	426.2
775	1427	268	514.4	218	424.4
750	1382	267	512.6	217	422.6
725	1337	266	510.8	216	420.8
700	**1292**	**265**	**509**	**215**	**419**
675	1247	264	507.2	214	417.2
650	1202	263	505.4	213	415.4
625	1157	262	503.6	212	413.6
600	**1112**	261	501.8	211	411.8
575	1067	**260**	**500**	**210**	**410**
550	1022	259	498.2	209	408.2
500	**932**	258	496.4	208	406.4
475	887	257	494.6	207	404.6
450	842	256	492.8	206	402.8
425	797	**255**	**491**	**205**	**401**
400	**752**	254	489.2	204	399.2
375	707	253	487.4	203	397.4
350	662	252	485.6	202	395.6
325	617	251	483.8	201	393.8
300	**572**	**250**	**482**	**200**	**392**
299	570.2	249	480.2	199	390.2
298	568.4	248	478.4	198	388.4
297	566.6	247	476.6	197	386.6
296	564.8	246	474.8	196	384.8
295	**563**	**245**	**473**	**195**	**383**
294	561.2	244	471.2	194	381.2
293	559.4	243	469.4	193	379.4
292	557.6	242	467.6	192	377.6
291	555.8	241	465.8	191	375.8
290	**554**	**240**	**464**	**190**	**374**
289	552.2	239	462.2	189	372.2
288	550.4	238	460.4	188	370.4
287	548.6	237	458.6	187	368.6
286	546.8	236	456.8	186	366.8
285	**545**	**235**	**455**	**185**	**365**
284	543	234	453.2	184	363.2
283	541.4	233	451.4	183	361.4
282	539.6	232	449.6	182	359.6
281	537.8	231	447.8	181	357.8
280	**536**	**230**	**446**	**180**	**356**
279	534.2	229	444.2	179	354.2

COMPARISON OF CENTIGRADE AND FAHRENHEIT DEGREES—(*Continued*)

Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.
178	352.4	128	262.4	78	172.4
177	350.6	127	260.6	77	170.6
176	348.8	126	258.8	76	168.8
175	**347**	**125**	**257**	**75**	**167**
174	345.2	124	255.2	74	165.2
173	343.4	123	253.4	73	163.4
172	341.6	122	251.6	72	161.6
171	339.8	121	249.8	71	159.8
170	**338**	**120**	**248**	**70**	**158**
169	336.2	119	246.2	69	156.2
168	334.4	118	244.4	68	154.4
167	332.6	117	242.6	67	152.6
166	330.8	116	240.8	66	150.8
165	**329**	**115**	**239**	**65**	**149**
164	327	114	237.2	64	147.2
163	325.4	113	235.4	63	145.4
162	323.6	112	233.6	62	143.6
161	321.8	111	231.8	61	141.8
160	**320**	**110**	**230**	**60**	**140**
159	318.2	109	228.2	59	138.2
158	316.4	108	226.4	58	136.4
157	314.6	107	224.6	57	134.6
156	312.8	106	222.8	56	132.8
155	**311**	**105**	**221**	**55**	**131**
154	309.2	104	219.2	54	129.2
153	307.4	103	217.4	53	127.5
152	305.6	102	215.6	52	125.6
151	303.8	101	213.8	51	123.8
150	**302**	**100**	**212**	**50**	**122**
149	300.2	99	210.2	49	120.2
148	298.4	98	208.4	48	118.4
147	296.6	97	206.6	47	116.6
146	294.8	96	204.8	46	114.8
145	**293**	**95**	**203**	**45**	**113**
144	291.2	94	201.2	44	111.2
143	289.4	93	199.4	43	109.4
142	287.6	92	197.4	42	107.6
141	285.8	91	195.8	41	105.8
140	**284**	**90**	**194**	**40**	**104**
139	282.2	89	192.2	39	102.2
138	280.4	88	190.4	38	100.4
137	278.6	87	188.6	37	98.6
136	276.8	86	186.8	36	96.8
135	**275**	**85**	**185**	**35**	**95**
134	273.2	84	183.2	34	93.2
133	271.4	83	181.4	33	91.4
132	269.6	82	179.6	32	89.6
131	267.8	81	177.8	31	87.8
130	**266**	**80**	**176**	**30**	**86**
129	264	79	174.2	29	84.2

COMPARISON OF CENTIGRADE AND FAHRENHEIT DEGREES—(*Continued*)

Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.	Degrees Centigrade.	Degrees Fahrenheit.
28	82.4	8	46.4	—12	10.4
27	80.6	7	44.6	—13	8.6
26	78.8	6	42.8	—14	6.8
25	**77**	**5**	**41**	**—15**	**5**
24	75.2	4	39.2	—16	3.2
23	73.4	3	37.4	—17	+ 1.4
22	71.6	2	35.6	—18	— 0.4
21	69.8	+ 1	33.8	—19	— 2.2
20	**68**	**0**	**32**	**—20**	**4**
19	66.2	— 1	30.2	—21	— 5.8
18	64.4	— 2	28.4	—22	— 7.6
17	62.6	— 3	26.6	—23	— 9.4
16	60.8	— 4	24.8	—24	—11.2
15	**59**	**—5**	**23**	**—25**	**13**
14	57.2	— 6	21.2	—30	—22
13	55.4	— 7	19.4	—35	—31
12	53.6	— 8	17.6	—38	—36.4
11	51.8	— 9	15.8	**—40**	**—40**
10	**50**	**—10**	**14**	—100	—148
9	48.2	—11	12.2	—200	—328

—274° C. (absolute zero) = —461° F.

WHOLESALE PRICES CURRENT

Of Various Substances Many of Which are Used in Lubrication November 1913

Mineral Oils

Black reduced, 29 gravity B., 25 @ 30 cold test	per	gallon	$0.12½	@	$0.13
29 gravity B., 15 cold test	"	"	.13	"	.13½
27 gravity B., zero cold test	"	"	.17	"	.17½
Summer	"	"	.12½	"	.13
Cylinder, light, filtered	"	"	.20	"	.20½
Dark, filtered	"	"	.17½	"	.18
Extra cold test	"	"	.24½	"	.25
Dark, steam refined	"	"	.14	"	.14½
Natural, Franklin, 32 gravity B	"	"	.11½	"	.12
Smith Ferry, 32 @ 34 gravity B	"	"	.12¼	"	.12¾
West Virginia, 29 gravity B	"	"	.20	"	.20½
Neutral filtered lemon, 33 @ 34 gravity B	"	"	.16	"	.16½
White, 32 @ 34 gravity B	"	"	.24½	"	.25
32 @ 34 B. gray, bloomless	"	"	.15¼	"	.19
32 B. gray, wool grade	"	"	.14	"	.15

Mineral Oils—(*Continued*)

Paraffin, high viscosity	per gallon	$0.24	@	$0.26
0.903–0.907 sp. gr	" "	.14	"	.14½
0.903 sp. gr	" "	.13	"	.13½
0.883 sp. gr	" "	.10½	"	.11
0.875 sp. gr	" "	.10	"	.10½
0.865 sp. gr	" "	.10	"	.10½
Red paraffin	" "	.13	"	.13½
Spindle No. 1, filtered	" "	.17	"	.18
No. 1	" "	.14	"	.15
No. 2	" "	.13½	"	.14
No. 3	" "	.12½	"	.13
No. 4	" "	.11½	"	.12

Vegetable Oils

Linseed, Domestic raw, in bbls	per gallon	$0.77	@	$0.78
Boiled, in bbls	" "	.78	"	.79
Calcutta, raw, in bbls	" "	—	"	.85
Rape-seed, in bbls. burning	" "	.72	"	.76
Blown	" "	.68	"	.70
Refined	" "	.64	"	.66
Cottonseed, yellow summer, prime	per pound	.06	"	.08
White summer	" "	.06	"	.07
Yellow winter	" "	.08	"	.09
Corn oil	" "	.07	"	.08
China wood oil	" "	.06¾	"	.07
Olive, yellow	per gallon	.80	"	—
Soap stock	per pound	—	"	.05¼
Palm, red	per cwt.	5.45	"	—
Lagos	per pound	.05¾	"	—
Cocoanut, Ceylon	" "	.07⅛	"	.07¼
Cochin	" "	—	"	.07⅛
Castor, No. 3	" "	.09½	"	.10½
Rosin, first run	per gallon	—	"	.21
Second run	" "	—	"	.23
Third run	" "	—	"	.25
Fourth run	" "	—	"	.27
Pine oil	" "	.30	"	.48
Soya bean oil	per pound	.06⅛	"	.06½
Tar oil	per gallon	.30	"	.31

Animal and Fish Oils

Whale, natural winter	per gallon	—	@	$0.44
Bleached winter	" "	—	"	.46
Extra bleached	" "	—	"	.48
Sperm, natural winter	" "	—	"	.76
Bleached winter	" "	—	"	.79

Animal and Fish Oils—(*Continued*)

Lard, ex. prime city, winter	per gallon	—	@	$0.88
Prime	" "	$0.56	"	.58
No. 1 extra	" "	.57	"	.58
No. 1	" "	.48	"	.50
Menhaden, prime crude	" "	—	"	—
Prime southern	" "	—	"	.23
Light strained	" "	—	"	.33
Bleached winter	" "	—	"	.36
Cod, domestic, prime	" "	—	"	.38
Newfoundland	" "	—	"	.40
Degras, American	per cwt.	2.15	"	2.25
French	" "	2.30	"	2.63
Neats-foot, 20° cold test	per gallon	.86	"	.87
30° cold test	" "	.81	"	.82
40° cold test	" "	.66	"	.67
Prime	" "	.55	"	.56
Tallow (acidless)	" "	.57	"	—
Prime	" "	.54	"	.55
Red, saponified	per pound	.05¾	"	.06¾
Elaine	per gallon	.43	"	.47

Wax, Tallow, Etc.

Paraffin wax, crude	per pound	$0.04½	@	$0.04¾
Refined, 120 @ 140 M. P	" "	.06¼	"	.13½
Japan wax	" "	.12	"	.12¼
Carnauba wax	" "	.32	"	.50
Bees wax, Yellow	" "	.32½	"	.34
Bayberry or myrtle wax	" "	.25	"	.26
Ceresin wax (yellow)	" "	.21	"	.30
Spermaceti	" "	—	"	.30
Stearine, lard	" "	—	"	.11½
Tallow	" "	.06	"	.07½

Apparatus for the Examination and Study of the Behavior of Valve and Cylinder Oils and Other Petroleum and Lubricating Oils in Saturated and Superheated Steam, Carbon Dioxide, Air and Other Gases[1]

By P. H. Conradson

Chief Chemist Galena-Signal Oil Company, Franklin, Pa.

The introduction of a high degree of superheated steam in locomotive and stationary engine practice has brought about the necessity of adding tests to the ones usually made of cylinder oils, whether straight petroleum stock, or mixture of the same with smaller or larger amounts of saponifiable fats.

The writer has constructed and used an apparatus for some time in the investigation of cylinder oils suitable for the lubrication of valves and cylinders in connection with the use of highly superheated steam in locomotive service.

The apparatus is essentially as follows (see photographic cut).

A, small steam boiler with gas, steam gauges and pipe connections.

B, superheater with armored thermometer, T, and steam connections (a small circulating water heater with double copper coils, answers very well).

C, sight feed lubricator with connections.

D, steam vessel (a conical cast iron retort for distilling mercury, about 95 mm. deep and about 75 mm. in diameter at top provided with two strong screw clamps to keep the lid tight, answers very well) surrounded on the outside, as indicated, by a coil of copper pipe 5–6 mm. inside diameter, for further heating of the steam and gases used in the tests; this coil is tapped into the bottom of the steam vessel containing a suitably supported dish of about 25 cc. capacity (an ointment box about 55 mm. in diameter, 18–20 mm. high) into which the oil to be examined is fed drop by drop from the sight feed lubricator C (or, a given amount of oil is poured into the cup I). Through the lid is inserted an armored thermometer, T, reaching down into the

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[1] Paper presented at the Eighth International Congress of Applied Chemistry, New York, September, 1912.

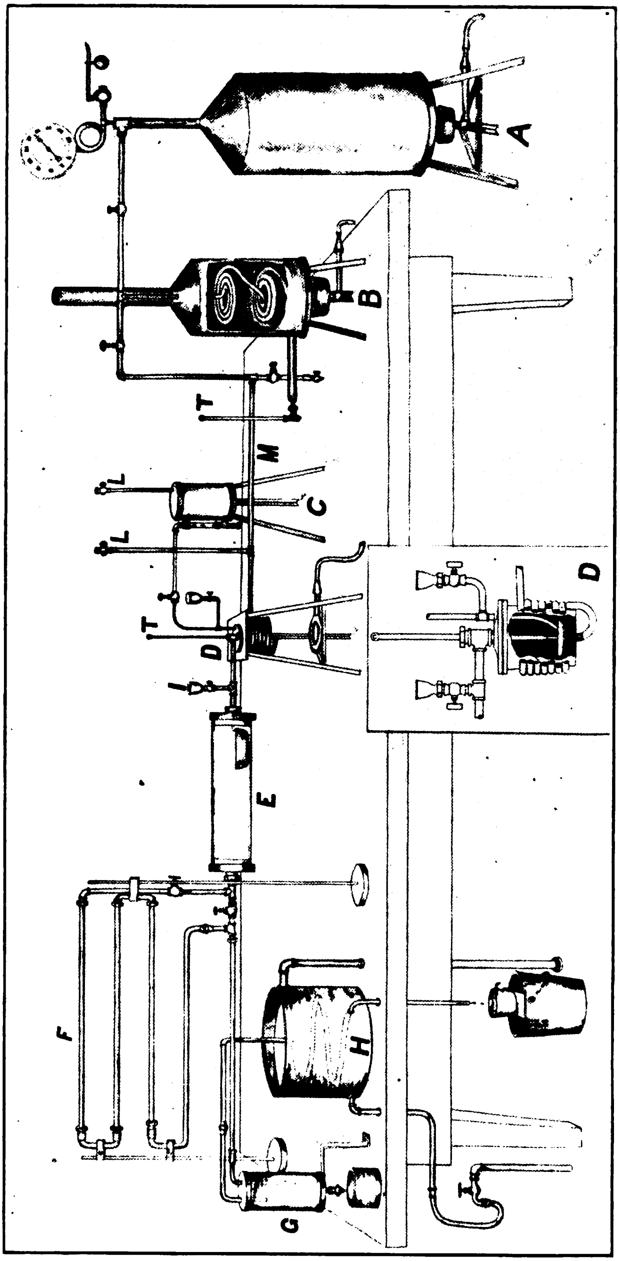

Fig. 116.

dish. The steam vessel thus arranged and connected is surrounded and covered by a suitable asbestos hood.

L, pipe connections for the gases used.

E, heavy glass cylinder about 200 mm. long and 18–20 mm. inside diameter containing a small rectangular boat or dish; beyond E is a glass tubing system, F, connected up as shown with brass fittings; each glass tube is about 500 mm. long and 8–9 mm. inside diameter; at the further end is attached a steam separator and oil collecting vessel, G, consisting of an ordinary glass chimney fitted up with brass caps; to this condenser H is connected by means of copper tubing.

In this apparatus any desired temperature may be maintained at least up to 1,000° F.; this is 300°–400° higher than is required under the most exacting service conditions as far as superheated steam temperature is concerned.

In studying the behavior of valve and cylinder oils in an atmosphere of superheated steam, the steam is passed from the small steam boiler through the superheater B and into the steam vessel D through its pipe coil (lamp being lit under D), and out through E, F, G and H.

When the temperature reaches and is regulated in D to 400° F., oil is fed drop by drop from sight feed lubricator C (or the cup I is filled); the steam is shut off and the valve in C (or I) opened, letting the oil run into the dish in D; the steam valve is then very gradually and carefully opened so as to let through any desired amount of steam.

The temperature in D is maintained for a given length of time at or near 400° F., noting if any oil vapors are coming over; by shutting and opening the steam valve it is easy to see if any vapors are coming over with the steam.

The temperature is increased in increments of 25°–50° F., at a time, until the desired temperature is reached, noting the behavior of the oil vapors passing on with the steam through the glass pipe system, etc. It is interesting to note that cylinder oils containing rather a large percentage of saponifiable fats or fat oils generally come over at much lower temperatures than the main portions of the petroleum stock oils that are commonly used in compounding first class cylinder oils.

In further study of the behavior of oils, the boat in the glass cylinder E may be filled through the cup I to the left, and subjected at variou temperatures to slow currents of superheated steam, carbon dioxide and air or mixtures of the same. The glass cylinder E during the last test is surrounded by a heavy asbestos covering to prevent radiation.

The cylinder oils may leave a residue in the dish in D at steam temperatures below 700° F.; if so, such residue should give a clear solution in 90 cc. of 0.65 sp. gr. (87° B.) petroleum ether (Pennsylvania) and show no precipitate on standing. At steam temperatures of 850°–900° F., all the oil has usually volatilized with the steam; good oils should leave no carbonaceous or coky residue.

While this apparatus was designed principally for the study and behavior of valve and cylinder oils in atmospheres of steam at various temperatures, it can be adapted very readily for the study of other oils at various temperatures in currents of air, carbon dioxide or other gases or mixtures of same with and without steam.

The accompanying table of comparative tests of six samples of cylinder oils, A, B, C, D, E and F, a petroleum distillate of interest in connection with the study of cylinder oils in superheated steam:

Hot Air Test = 13 grams oil in shallow, round, flat bottom, iron dishes exposed six hours at 540° F. in a specially designed air bath.

Gasoline Test = 10 cc. oil, 90 cc. petroleum ether 0.65 sp. gr. (from Pennsylvania crude) in graduated flat precipitating tubes, taking reading after one hour's standing.

Carbon Test using 35 grams Oil, according to Conradson's apparatus and method.

Superheated Steam Test = 13 grams of oil used.

Sample "A" in a superheated steam test at 800° F. (427° C.)

	A	B	C	D	E	F
Flash point (open cup)	545° F.	550° F.	605° F.	595° F.	550° F.	365° F.
Burning point	610° F.	630° F.	695° F.	680° F.	630° F.	415° F.
Gravity at 60° F. Baumé	26.4	26.1	24.7	25.9	—	33.5
Sp. gr. at 15° C	0.895	0.897	0.905	0.898	—	0.856
Color	Light	Dark	Dark	Very dark	—	Yellow
Gas, test before fire test	Good	Good	0.5 cc.	4.0 cc.	—	—
Gas, test after fire test	Good	Good	Good	3.0 cc.	—	—
Cold test flows	+55° F.	+32° F.	+45° F.	+34° F.	+30° F.	Zero
Saponifiable fats	Trace	Trace	Trace	Trace	15%	None
Vis. Saybolt at 212° F. (60 cc.)	133 sec.	146 sec.	215 sec.	216 sec.	—	—
Barbey Ixomet 500° F. (180 units = 30 cc.)	45 sec.	51 sec.	61 sec.	60 sec.	—	—
Hot air test at 540° F. (loss)	15%	11%	2.5%	5.5%	—	—
Gas, test after	Good	Good	0.5 cc.	3.5 cc.	—	—
Carbon test residue	2.15 %	2.70%	4.90%	5.10%	—	—
SO_3 in residue	0.023	0.03	0.03	0.04	—	—
Loss in superheated steam at 400° F.	0.0	0.0	0.0	0.0	0.0%	32.0%
500° F.	5.0	4.0	1.5	1.0	5.0	67.0
600° F.	18.5	18.0	6.5	8.5	21.5	—
700° F.	44.0	34.0	32.5	40.5	—	—
Total loss up to 700° F.	67.5 %	56.5 %	40.5 %	50.0 %	26.5%	99.0%
Gas test of oil: Residue from 700° test	Good	Good	1.5 cc.	3.0 cc.	—	—

left no residue. Sample "C" left 2.5 per cent. dry carbonaceous residue.

Sample "E" containing 15 per cent. of saponifiable fats subjected to the superheated steam test lost 26.5 per cent. up to 600° F.; the oil residue from this test contained 17.5 per cent. saponifiable fats; this indicates that the petroleum oil stock ("B" used) goes off with the steam somewhat faster in proportion to the fat oil up to 600° F. (350° C.).

The steam pressures used in these tests were about 10 to 12 pounds per sq. in. A large volume of superheated steam passed through the apparatus during the tests (about 40 cc. condensed steam per minute).

In these superheated steam evaporating tests about 13 grams of oil were weighed into the small dish placed inside the steam vessel D. (The capacity of the small iron dish is 50 cc., having a diameter of about 48 mm. and 30 mm. high, with flat bottom.)

The steam vessel with the oil in the dish was heated up to about 350° F. (176° C.), passing a slow current of natural gas through the apparatus, then superheated steam was admitted, the gas shut off, and the temperature raised up to the required degree and kept constant for about 75 minutes; the volatile matter in the oils at the given test temperature generally were carried over with the steam inside of 60 minutes, allowing about 15 minutes' extra steaming. At the end of each given temperature test the steam and heat were shut off; after cooling, the dish containing the oil was weighed and replaced in the steam vessel and the operation repeated for the next temperature test.

GALENA-SIGNAL OIL COMPANY,
Franklin, Pa.

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